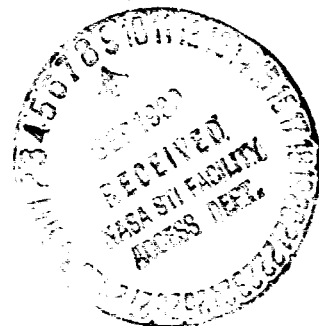


## Potential Release of Fibers From Burning Carbon Composites

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## **POTENTIAL RELEASE OF FIBERS FROM BURNING COMPOSITES**

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### **SUMMARY**

A comprehensive experimental carbon fiber source program was conducted to determine the potential for the release of conductive carbon fibers from burning composites. Laboratory testing determined the relative importance of several parameters influencing the amounts of single fibers released, while large scale aviation jet fuel pool fires provided realistic confirmation of the laboratory data. The dimensions and size distributions of fire-released carbon fibers were determined, not only for those of concern in an electrical sense, but also for those of potential interest from a health and environmental standpoint. Fire plume and chemistry studies were performed with large pool fires to provide an experimental input into an analytical modelling of simulated aircraft crash fires. A study of a high voltage spark system resulted in a promising device for the detection, counting, and sizing of electrically conductive fibers, for both active and passive modes of operation.

### **INTRODUCTION**

The disclosure (ref. 1) of the possibility for widespread damage to electrical and electronic equipment due to the inadvertent release of conductive carbon or graphite fibers into the atmosphere resulted in the formation of a carbon fiber risk analysis program with the objective of assessing the potential risk in detail (ref. 2). The initial element of the NASA study was that which required a study of the source of the fiber itself. Since the NASA program was to be concerned primarily with risks resulting from the use of carbon fibers in civilian aeronautical applications, the most likely source of carbon fibers was assumed to be from the combustion of carbon fiber-reinforced composite aircraft parts. At the beginning of the program, and to the present time (1980), no instance was known of a crash and/or fire involving any of the few commercial transport aircraft having carbon fiber composites in active service. However, several such crashes and fires involving military aircraft with carbon fiber composites have occurred, which together with a body of accident statistics for commercial aviation suggest the inevitability of accidental carbon fiber release from civilian aircraft crash fires.

Without experience to draw on, it was necessary to develop and execute a test program to provide the data for predicting how much and what kind of fibrous materials might be released from future aircraft crashes. Since the fiber source element was to be the first input into the comprehensive risk analysis, time was important in developing the tests. Only sparse knowledge of fire release was available at the start of NASA's program. Limited qualitative laboratory tests had been carried out at the Naval Research Laboratory (ref. 3). Those tests were principally thermogravimetric analysis and fire tests on very small composite specimens. The results disclosed some important effects on the burning of composites. For example, the formation of a carbonaceous binder, or char, from the burned epoxy resins was helpful in holding the residual carbon fibers together. Furthermore, it was noted that some form of agitation, intentional or otherwise, was instrumental in causing the release of single fibers.

Pioneering studies in fiber release from burned composites were conducted at the Naval Surface Weapons Center, Dahlgren, Virginia (ref. 3). The tests were run in an enclosed room, which meant the complete containment of all fiber materials given off during the test. The test procedure involved the combustion of composite specimens up to 0.1 m<sup>2</sup> (1 ft.<sup>2</sup>) in size with a propane-air flame. Most of the early tests run by the Navy prior to NASA's entrance into the carbon fiber study involved the destruction, subsequent to the fire, of the composite panel with 57 grams (2 ounces) of explosive placed beneath the specimen. Naturally, that test sequence resulted in an extensive release of a variety of fibrous materials (fig. 1). The residues were collected on an array of more than a hundred sheets of paper coated with a tacky adhesive to retain the fibers when they fell onto the papers. The count of single fibers, which were considered to be the most likely form of fiber to cause electrical damage over a widespread area, was supplied by a group at the U. S. Army's Dugway Proving Ground, Utah. Initially, the amounts of single fibers released from the burning and exploding of flat composite plates were thought to be very high, as much as 20-25% of the mass of carbon fiber originally present in the specimens. Later, corrections and refinements to the counting procedure showed that the amounts of single fiber released were almost always less than 10%.

The afore-described test sequence was intended to simulate the crash scenario for military aircraft. Based on prior experience, the burn plus explode test appeared to be valid for the most severe types of military aircraft crash events, in particular those involving crashing and/or burning on the deck of an aircraft carrier. However, the likelihood that the same test would simulate realistically the crash of a commercial air transport was thought to be very small. Nevertheless, the burn/explode test was applied at the beginning of the NASA program to several composite materials, including aircraft parts either of the type in

use or expected to be used on commercial aircraft. It was believed that such tests could be considered the ultimate in severity in crash situations. Following those tests were to be a large number of others, at a number of locations, which would prescribe test conditions covering a wide range of expected commercial aircraft crash/fire events.

This report describes that test program and presents the total results of the testing which led to the amounts of carbon fibers which could be expected to be released accidentally from the crashes, and ensuing fires, of commercial air transports. Specific amounts of fibers were recommended for application to the risk analysis computations. In addition to the amounts of carbon fibers, the physical characteristics and dimensions of fire-released carbon fibers were determined and that knowledge was relayed to appropriate parties active in the federal carbon fiber study. Use of trade names in this report is for the sole purpose of identifying the materials tested, and such use does not constitute an official endorsement of the products, either expressed or implied, by NASA.

#### SCOPE OF TEST PROGRAM

The various facilities which have supplied data for the carbon fiber source test program have been summarized in Table I. The test program was under the overall direction of NASA Langley Research Center. In addition, an extensive investigation of the fundamentals of combustion of both epoxy resins and carbon fibers was performed at Langley. The initial testing, and eventually the major portion of the test program was conducted under NASA contract by Naval Surface Weapons Center personnel at Dahlgren, Virginia, using the previously-described burn/explode test procedure (ref. 4, 5). Those tests were then followed by a comprehensive program at the Navy facility which correlated the amount of single carbon fiber release with numerous factors involving the degree and type of disturbance to the composite residue during and after the fire (ref. 6). Another laboratory-scale carbon fiber release study was conducted by the AVCO Corporation, Lowell, MA, to provide some important fundamental composite combustion characteristics (ref. 7). Some of the earliest fiber-release data obtained under realistic agitation conditions was made available by Scientific Services, Inc., on a contract from NASA-Ames Research Center (ref. 8). The Jet Propulsion Laboratory carried out a program to develop techniques for detecting and quantifying released fibers (ref. 9), a continuing need throughout the carbon fiber risk analysis program.

Demonstration tests were carried out at three different locations. The demonstration tests were intended to establish the validity of laboratory-scale tests. All three test series were

successful in providing useful data about the extent of release of carbon fibers from realistically large, unconfined fuel fires, even though fiber release was not always the primary test objective. The first demonstration tests were conducted by the TRW Defense and Space Systems Group at the Naval Weapons Center, China Lake, CA, under Air Force sponsorship (ref. 10). NASA's role in those tests was the support of TRW for the reduction and analysis of a dozen outdoor composite burn tests, beginning with the burn/explosion of 0.1 m<sup>2</sup> composite plates and culminating in the outdoor burning of full-size composite aircraft parts. A second series of demonstration tests was conducted at the Naval Surface Weapons Center, Dahlgren, in a rather unusual test chamber, a 0.8 km (one-half mile) long steel tube, a 275-meter (900 feet) portion of which was used for the test (ref. 11). The principal objective of that test series was to expose electrical equipment, previously shown to be damaged by virgin carbon fibers in laboratory tests, to carbon fibers released by burning carbon composites with aviation jet fuel. The third set of demonstration tests was conducted at the Army's Dugway Proving Ground, with two main objectives: to determine the amount and nature of fibrous materials released when substantial quantities of carbon fiber composite parts were burned outdoors in a large-size jet fuel pool fire, and to monitor the downwind dissemination of fibrous products from the fire (ref. 12, 13, 14, 15, 16).

A series of tests which did not afford any direct fiber release data did, however, provide a body of fundamental knowledge about fires which was useful in explaining certain observations about the characteristics of released fibers and in confirming the amount of fiber which was consumed in large fuel fires. That program involved the study of several large pool fires at NASA's White Sands, NM, test range (ref. 17). The fires were instrumented extensively to determine the gaseous chemical species and their concentrations, and the temperatures and velocities within the fire plumes. The resulting data was then applied to the development of a model to describe the nature of large, liquid hydrocarbon pool fires.

## RESULTS AND DISCUSSION

### CARBON FIBER RELEASE FROM COMPOSITES

#### Dahlgren Chamber Tests

##### Burn Plus Explosion Tests

Composite aircraft parts.— A general description of the room in which the Dahlgren chamber propane burn/explode test was carried out is given in reference 3. Additional details of the experimental procedure, test equipment, the array of collection

papers, and the collection of all fiber residues is reported in reference 4. A Boeing 737 spoiler, identical to a number of such composite spoilers in flight service on commercial airliners, was the source of the test specimens. The 0.527 m by 1.32 m spoiler was fabricated with upper and lower composite skins of up to 6 (upper) and 8 (lower) plies of T-300 carbon fiber - 5209 epoxy tape. An aluminum frame with hinge fittings and some glass fabric was also present. The test spoiler, which had been failed in a flexural test as a cantilever beam, had a long fracture in a diagonal direction across the width of the spoiler and through the composite skin. The spoiler was cut into 15 specimens which were tested with the burn/explode procedure described in reference 4. In addition, three duplicate specimens were cut from a second spoiler and tested in a burn-only mode for comparison with the corresponding burn/explode test results.

Two portions of DC-10 carbon fiber composite rudder parts, fabricated from T-300 fiber - 5208 epoxy tape, were also cut into various sized test specimens. One part, called "rudder no. 1", did not have the glass fiber-epoxy leading and trailing edges which were included in the "rudder no. 2" part.

The results of those burn and burn/explode tests for which complete fiber count data was obtained are presented in Table II. Two sets of results have been given in the table. The uncorrected results for the numbers of fibers accounted for, weight of fibers accounted for, and weight percent of fiber release based on the amount of fiber calculated to have been present originally in the specimen were the original counting data from Dugway Proving Ground. The corresponding corrected results reflect adjustments made to the early data as a result of a round-robin counting study by four different laboratories (ref. 18) and a subsequent independent data analysis (ref. 19). Weighted averages for the spoiler and rudder results were determined by dividing the total weight of carbon fiber released from all tests by the total weight of carbon fiber initially present, and multiplying by 100.

The results show a wide variation from specimen to specimen in the amount of fiber release by burning and exploding small (225 to 1000 cm<sup>2</sup>) specimens of spoilers. The variations in the DC-10 rudder test results were much less extreme. In view of the different burn times for the spoiler tests, it is highly unlikely that the results can be reconciled with the test variables. Even tests with the same burn times but cut from different locations in the spoiler (e.g., BT-119/X-89 and BT-120/X-90) gave widely differing results. The scatter obtained from the spoiler tests could be attributed, in part, to several features of the construction of the spoiler. Certain specimens had different thicknesses of aluminum honeycomb between the two composite skins, which would have an effect on the insulation of the upper skin of the test specimen (mounted horizontally) from the flame. Different results

would also have been expected for specimens which included portions of the heavy metal frame or hinge fittings, in comparison to those with none of the solid metal pieces. The variations in results could have arisen by virtue of the heavy metal masses serving as heat sinks and thus moderating the thermal effect on the composite skin. Perhaps, too, the heavy metal masses from the hinge fittings could have either moderated, or magnified, the destructive effect of the explosive on the upper composite skin of the test specimens. There also may have been a direct correlation between the average fiber lengths and the weight percent of released fibers, but no attempt was made to determine how significant the relationship was beyond the fact that, naturally, the longer the fibers are, the more they will weigh.

Several other types of aircraft structural parts were exposed to the burn/explode test at Dahlgren (ref. 5). However, the expense and time required to count the fibers permitted the evaluation of only two tests besides the 737 spoiler and DC-10 rudder tests. In one instance, a 15 cm x 15 cm specimen of a 1-cm thick Nomex honeycomb panel with two 2-ply carbon-epoxy skins, used as flooring in some commercial airliners, was burned for 1200 seconds (selected as a standard burn time) and then destroyed with an explosive blast. The amount of single fiber released from the panel was nearly equivalent (5%) to the average amount released from the spoiler tests (5.6%). The same burn/explode test procedure applied to an experimental, high tip speed jet engine fan blade, fabricated at NASA's Lewis Research Center from AS carbon fiber and PMR-11 polyimide resin, produced only 1.1% single carbon fibers. This lower release may reflect a beneficial effect of the residual char remaining from the combustion of the polyimide resin.

Thickness and configuration effects.- Early in this fiber release test program, some signs were seen that certain factors such as the relative thickness of the burned composite might play a role in the amount of fiber released. Obviously, the manner in which a composite panel is "laid-up", or put together, that is, in unidirectional, cross-ply or woven configurations, could also be important from the standpoint of fiber release mechanism. A series of burn and burn/explode tests were run using panels with 1.6, 3.2, and 6.4 mm (1/16", 1/8", and 1/4") thicknesses for both unidirectional and cross-ply configurations (woven samples were not available). The tests were run using the same procedures as for the aircraft parts, except that the burn times were held constant at 1200 seconds and 15 cm x 15 cm specimens were used. Detailed results of those tests are tabulated in references 6 and 20, but a plot of the test results is presented in figure 2.

Figure 2 shows that the amounts of single fiber released when the specimens were burned only were much, much less than when burned and exploded. There appeared to be a slight trend toward greater fiber release for thin, cross-ply samples. The somewhat



higher release for 3.2 cm unidirectional specimens in both burn and burn/explode tests is thought to be due to the poor quality of those composite panels, which had only 25 weight percent resin versus 30% for the others. C-scan tests showed them to be porous, with large voids. It seemed reasonable that if portions of the fibers within the voidy areas had not been wet by the resin during the manufacturing process, then those fibers would be readily released when the composite burned. This was the first indication that the quality of composites might be a factor in the release of carbon fiber. However, stringent quality control procedures exercised in the manufacture of composite aircraft parts would prevent the use of such poor quality materials.

A summary of the amounts of single fibers released from all of the Dahlgren burn/explode tests is given in figure 3. The 6.4 mm thick NASA flat plate was the same 6.4 mm cross-ply panel shown in figure 2. The corresponding 6.4 mm NAVY flat plate was also a cross-ply composite, but the specimen was four times as large ( $0.1 \text{ m}^2$ ) as the NASA plate ( $0.025 \text{ m}^2$ ).

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#### Burn Plus Non-explosive Disturbance Tests

The burn/explode tests described above were, admittedly, far more destructive than could be imagined for the majority of civilian aircraft crashes. To answer that valid criticism, a comprehensive test program was run in the Dahlgren chamber (ref. 6). That program involved the disturbance of post-burned composite specimens in a number of ways which were felt to be more representative of the types of disturbing events in a commercial airliner crash-fire. All of the tests were performed by first burning standard composite specimens for 1200 seconds with the same propane-air flame used for all of the Dahlgren chamber tests. The 15 cm x 15 cm, 0.45 cm-thick specimens were cut from 60 cm x 120 cm composite panels. The panels were fabricated from 0.014 cm AS fiber/3501-6 epoxy resin pre-preg tape, with 24 plies laminated in a  $(0, \pm 45^\circ, 90^\circ)_3$  arrangement. The panels were uniformly excellent in quality.

Air blast and air flow.- Two sets of tests in the burn plus disturbance program employed conditions which were thought to be the extremes in the crash-fire of a commercial air transport. The first set of tests involved the instantaneous release of pressurized air such that the velocity of the air directed at the edge of the panel residue of the burned composite (after the 1200 seconds burn period) ranged from 70 to 244 meters per second (135-470 knots). The highest velocity simulated the force from an exploding fuel tank while 70 meters per second was considered representative of flame or gas velocities generated by a raging fire or "firestorm".

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The second set of tests involved directing a flow of air,

5 and 15 meters per second (10 and 30 knots) for 600 seconds at the edge of the test specimens either during or immediately after the routine 1200 second burn period. Those levels of disturbance were thought to be typical of the influx of air into a jet fuel pool fire, but could also be typical of winds affecting the residual fibrous residues after the composite parts had been burned.

Reference 6 reports the specific details and individual test results for both the air blast and air flow test series. A summary of the results of both types of tests is presented in figure 4. The amounts of single fiber released by a 600-second low velocity flow (5 m/sec) of air, both during and after the combustion of the composite, were relatively low. The extremely low amount of fiber given off when the 15 m/sec air flow was applied to the burning composite, in contrast to the nearly 1% given off after the burn was complete, was due to the fact that the composite was kept cooled down by the air flow, thus preventing the burn-off of the resin. However, both situations seem to be likely occurrences in a crash-fire. The release of 2½ to nearly 4% single fiber as the result of the impact of high pressure air also seems to be valid for events that could occur in the most severe types of commercial aircraft crash fires. Although explosions such as those from ruptured fuel tanks, according to accident records, are the exception rather than the rule, they do happen in about 15% of the crashes and must be accounted for in the risk analysis. Obviously, they can approach ordnance-type explosions in severity from the standpoint of promoting release of single fibers (c.f., fig. 3).

External (pendulum) impact.- The simulation of the effect of mechanical impacts, as could result when heavy metal parts were either dropped or propelled against burned composite parts, was accomplished using a pendulum impact tester. A complete description of the test apparatus and procedure, as well as complete test results, has been reported in reference 6. Five different pendulum heads were used for the tests, with the burned composites being impacted by the pendulum heads at four different angles. A summary of the results, presented in figure 5, shows that all combinations gave off less than one-quarter percent single carbon fiber, based upon the amount of fiber initially present in the composite specimen. These results, together with others to be given later in this report (drop impact tests), suggest that disturbances such as those caused by the collapse of portions of aircraft structures (e.g., a vertical stabilizer falling onto a burning or burned composite horizontal stabilizer) would have a minimal effect upon the amount of single fiber released.

Internal disturbances.- If the types of disturbances to the burned composites described above are considered to be externally caused (explosions, or impacts by air or mechanical objects), then another general form of disruption of the burned remnants could conceivably be generated by forces internal to the composites. Such forces as twisting, flexing, vibrating or dropping could be

caused by the heating or cooling of the burning or burned composites, or from such actions as the breaking off or twisting off of portions of composite aircraft structures. A series of tests to determine the severity of internal forces in bringing about the release of single carbon fibers was conducted in the Dahlgren chamber and is reported in detail in reference 6.

As is apparent from the summary of results in figure 6, all of the internal forces studied (flexing or twisting the composite remnants to destruction, vibrating at 30 hertz, or dropping the burned composite from a height of 2.44 meters - all after the standard 1200 second burn period) generated amounts of single fibers equally as small as were released by the pendulum impact tests. Therefore, it was concluded that those actions, which could certainly prevail in raging fires, would contribute an insignificant amount to the total release of single carbon fibers.

#### Burn Testing of Honeycomb Core Panels

Among the aircraft parts tested by burning and exploding the specimens was a 1-cm thick panel composed of a Nomex honeycomb core and two carbon/epoxy skins. The burn plus explode tests showed (figure 3) that about as much single fiber was released from that panel as from the average of the 737 spoiler test specimens, and slightly more than from the averaged DC-10 rudder specimens. It was later found that the two-ply carbon/epoxy tape skins were covered on the outside with a thin glass fiber scrim cloth.

When tests less severe than the burn-explode test were conducted on the honeycomb panel, the results were rather unusual. Simply burning the panels with a propane-air flame in the Dahlgren chamber released an extremely small amount of fibers: about 0.01% of the mass initially present in the specimens. However, the application of 15 m/sec air flow at the fibrous residue (after a standard 1200 seconds burn time) released nearly 2% single fibers, while a 244 m/sec blast of air directed at the post-burned fibrous residue released 8% single fibers. Perhaps more important than the amounts of fibers released was the nature of the fibers. Those given off under burn conditions only had a mean length of 2.8 mm (for all fibers over 1 mm in length). Those given off as a result of air flow and air blasts were abnormally long: from the ten-minute flow of 15 m/sec (30 knots) air, the fibers averaged 8.3 mm, while those given off as a result of the instantaneous blast of high pressure air had an average length of 5.7 mm. In both cases, there were some very long fibers, that is, 20-50 mm long, a phenomenon which had not been observed in other tests.

The honeycomb panel test results injected a question into the matter of carbon fiber release from composite aircraft structures. While the weight percent or mass of released single fibers (8%) was fairly high, the absolute numbers of fibers were lower, naturally, because of the greater mean lengths. On the other hand, the fibers from the honeycomb panels were considerably longer than had been found in burn tests of other materials, which generally gave

off fibers with mean lengths between 2 and 3 mm. The vulnerability of electrical equipment to carbon fibers has been shown to increase with increasing fiber length (ref. 21), so if the release of abnormally long fibers from all such carbon fiber-honeycomb panels was typical, it might be of some concern in terms of the potential risks.

Accordingly, two other types of honeycomb panels of the same general configuration were obtained for study. One such panel had skins consisting of two plies of carbon fiber woven fabric impregnated with epoxy resin, while the other type of panel had skins composed of one ply of carbon fabric and one ply of Kevlar fabric, both impregnated with epoxy, with the carbon fiber on the outside of the panel. Both panels had a 1.27-cm thick Nomex honeycomb core. These two types of finished panels, as well as the one tested earlier, are expected to be used extensively in future composite aircraft structures. Specimens of each, 15 cm x 15 cm, were examined by means of the burn only, burn plus 15 m/sec. air flow, and burn plus 244 m/sec. air blast procedures. Individual test results are given in reference 6. A summary of the test results for all three types of panels is given in figure 7.

The application of a post-burn, 15 m/sec. flow of air to the burned composite panel with carbon fiber fabric alone (B) released more single fibers (4%) than from either panel A (unidirectional carbon fiber tape with glass fiber scrim cloth) or panel C (single layers of carbon and Kevlar cloth). However, the fibers which were released from panel B were much shorter than from panel A. The post-burn air blast test gave much less single fibers and of shorter length from panel B than from panel A. The amount of fibers given off by the carbon/Kevlar hybrid panel (C) was almost as great as by panel A, but the mean fiber length was considerably less.

In contrast, the summary of the burn only tests for the three panels at the right side of figure 7 (note the different scale) shows that panel A released much less single carbon fiber than did panels B and C.

Observation of the actual burn only tests suggested that the presence of the glass fiber scrim cloth overlaying the burning carbon fiber tape skins of panel A seemed to prevent the release of single fibers in the absence of a strong disturbing force. However, the scrim cloth also seemed to protect the underlying long, unidirectional carbon fibers from oxidation so that when the strong flow or blast of air was applied, unusually long fibers were available for release when the glass scrim cloth was blown away. Obviously, the use of carbon fabric skins alone and hybrid carbon/Kevlar fabric skins was effective in reducing the lengths of the released fibers, although it did not necessarily diminish the mass of carbon fibers given off. Although a complete correlation of the effects of the various factors, individually and collectively, upon the potential

risk of damage to electrical equipment were not completely correlated, the conclusion was reached that the future use of the honeycomb panels tests do not present a significantly greater threat to electrical equipment than the other types of carbon composite materials which were tested earlier.

#### Burn-Only Testing of Carbon Materials

The extreme event for releasing minimum amounts of carbon fibers during the burning of composites was considered to be the simple burning of the composite with no intentional disturbance during or after the fire. For several of the series of tests discussed above, simple burn tests alone were run on composite specimens in the Dahlgren chamber to provide a contrast to the destructive burn/explode tests. Some of the "burn only" test results were previously given in figures 2 and 7. Figure 2 shows that 4 out of 5 flat composite panels of varying thickness emitted less than 0.1% single fibers when simply burned alone with the circular propane-air burner used in the Dahlgren chamber. The lone exception was a 3.2 mm specimen of doubtful quality (resin-poor, porous) which released only 0.2% single fibers. Likewise, figure 7 shows that the amounts of single fibers released from burn-only tests on three different carbon fiber skin-honeycomb panels were equally small, about 0.2% or less, in contrast to up to 8% fibers released when the burned composite specimens were destroyed by high velocity air blasts.

Further evidence of the need for a disturbance to cause a significant release of fibers was afforded by the simple burning of three pieces of 737 spoiler, which were identical in size, shape, and location from the spoiler to three which had previously been burned and exploded. In this set of tests, the amounts released from the three burn only tests (figure 8) were less than 0.01% single fibers based on the weight of carbon fiber initially present in the specimen. Perhaps the dramatically lower fiber release from the burn only tests can be attributed to the presence of the metal portions of the test samples, which might have acted as heat sinks and moderated the thermal environment of the samples during the fire.

Two samples of virgin (raw) carbon fiber, wound as continuous tow on plastic spools, were burned to determine how much might be released from a mass of fiber previously uncombined with resin. A 0.45 kilogram spool of T-300 fiber released a barely detectable amount of single fibers, 0.0005%, after the standard 1200 second burn period; a 0.63 kilogram spool of HMS fiber gave off 0.01% of singles. More interesting than the amounts released was the observation that the residual mass of T-300 fiber after removal of the flame continued to glow for 5400 seconds, resulting in a total mass loss of more than 90% due to oxidation of the amorphous T-300 fiber. However, when the flame was removed from the HMS fiber mass, burning ceased immediately, and no mass loss could be detected. HMS

fiber is considered to be more "graphitic" in structure than T-300, or at least has a more ordered carbon structure, because of the higher processing temperatures used during its manufacture. Consequently, HMS is expected to be more thermooxidatively stable than T-300.

A summary of all the burn-only test results for three specific 737 spoiler specimens and a type A (see fig. 7) carbon fiber-fiber-glass skin, Nomex honeycomb core panel, together with the amounts of carbon fibers released when the same type specimens were subjected to burn plus explode tests, is given in figure 8. Also given are the results for the burn-only tests of the virgin carbon fibers on the spools, which had not previously been combined with a resin to form composites. Those results indicate that a fire involving raw carbon fibers, such as might occur in a storage warehouse or during a manufacturing operation, would probably release relatively small amounts of electrically hazardous single carbon fibers.

#### AVCO Fire Test Facility

A fire simulation test facility which had been built previously by the AVCO Specialty Materials Division, Lowell, Massachusetts was put to good use in the carbon fiber release program. The existing facility, which had been used to study the development of fire protection materials, utilized natural gas as the convective heat source. In addition, it had a radiant heating system which helped make the system attractive for simulating the burning of composites in jet fuel fires, since realistic combinations of radiant and convective heat fluxes could be obtained.

The existing chamber was modified in several ways which made it more suitable for studying composite burning and carbon fiber release. First, the chamber was modified to supply a forced air draft up to 8 meters/second through the test section. Air fuel mixtures ranging from 6:1 to 20:1 were then possible. A method of agitating the fibrous residue during and after the combustion of the composite specimens (nominally 5 cm x 12.5 cm in size) was provided for with a pulsating flow (5 pulses/second) at about 5 m/second of argon gas directed at the sample. And lastly, a fiber trapping chamber was assembled at the end of the fire test section. This chamber utilized a falling water curtain which efficiently trapped the fibers which were released from the burning composite. The water containing the entrapped fibers was filtered to catch the fibers and other particulate matter, mostly soot. When a fine spray of detergent solution was applied to the wet filter, the finely divided soot became wetted and passed through the somewhat porous filter material, leaving the fibrous material behind, to be measured gravimetrically. Complete details of the construction and operation of the improved test facility are reported in reference 7.

The AVCO fire test facility was used for a fundamental study of some variables which are important in the potential release of carbon fibers from burning composites. Three of the variables studied gave the results shown in figure 9. Variation of the fuel-to-air ratios from lean to rich, holding other variables constant, did not have a marked effect on the amounts of fibers that were given off, although it did make a considerable difference in the amount of fibers which were consumed, or oxidized away by the fire. However, the effect of edge restraint in reducing the amounts of fibers released was clearly demonstrated, since nearly ten times as much fiber was given off from a specimen with three raw edges exposed to the flame as evolved from a specimen which was mounted in a rectangular metal frame. That could be a significant factor in the release of fibers from shattered composite parts of a crashed aircraft. Although the AVCO facility employed additional air flows of up to 8 m/sec. (15 knots) past the burning test specimen, pulses of argon directed at the burning composite plates were used to simulate turbulent forces in a raging fire. The results in figure 9 show that the argon pulses did, indeed, increase the amount of released fibers.

A study of the effects of fire variables on the extent of oxidation, or burning up, of carbon fibers was also performed by AVCO and those results have been presented in a later section of this report on the characteristics of fire-released carbon fibers. Full details of the AVCO study are reported in reference 7.

#### Scientific Services, Inc. Test Facility

One of the earliest fiber release test operations was the drop impact chamber developed by Scientific Services, Inc. at Redwood City, CA under contract NAS2-9945 from NASA's Ames Research Center. Tests in that facility provided some very early fundamental composite burn results which were of value to the carbon fiber risk analysis program.

The test procedure involved the burning of 0.1m<sup>2</sup> composite panels with an array of highly radiant, natural gas burners. Following the combustion of the composites, projectiles of various weights were dropped from selected heights onto the fibrous residues of the specimens. In order to allow for subsequent analysis, a cover immediately encased the impacted specimen and the fibrous remnants which had been given off. Full details and results of that test program were reported in reference 8. Figure 10 shows some typical results, both of the amounts of single carbon fibers released and of total fragments ejected from the burned composite by the impact. No significant difference in the amounts of single fibers emitted were noted, either for different drop weights and heights or for woven carbon fabric used in the panel construction versus crossplied fiber tape. However, up to a ten-fold reduction in the amounts of fragments resulted from impacting the burned

composites made from carbon cloth, compared to those fabricated from crossplied carbon fiber tape. Visual observation disclosed that a relatively "clean" hole had been punched through the woven sample in contrast to a gross shattering of crossplied specimens. This suggests that woven construction composite parts on aircraft would be more resistant to damage by impacts from projectiles or other articles than would those fabricated from unidirectional tape.

#### TRW Outdoor Tests - China Lake

The first large-scale outdoor tests designed to release carbon fibers from burning composites were conducted in 1978 by the TRW Defense and Space Systems Group at the Naval Weapons Center (NWC), China Lake, CA. The tests were run under a contract from the Air Force's Rome Air Development Center, Griffiss Air Force Base, NY, while the test data was reduced, analyzed, and reported through a contract from NASA (ref. 10). The general objectives of the test series were: (1) to verify the results of the closed chamber composite burn-explode tests which had been conducted by the Navy at Dahlgren, VA, and (2) to simulate aircraft accidents in which carbon fibers might be released.

#### Test Procedures

A summary of the tests in the program has been given in Table III. The first three tests, which were preliminary to the NWC-China Lake tests, were actually carried out at TRW's Capistrano Test Site (CTS) at San Clemente, CA. Three 0.1 square meter x 6.4 mm thick carbon fiber composite (T-300/5208) plates were each burned with a propane flame after which the remaining fibrous material was destroyed with an explosive blast. The entire test was conducted in a 4.57 m x 4.57 m tent-like enclosure with 1 mm mesh screen sides and top. Instrumentation devices, both passive and active, were placed throughout the enclosure to monitor the fibers released. The three CTS burn-explode tests served well as preparation for the subsequent outdoor burn-explode tests at China Lake.

NWC tests 4, 5, and 6 were outdoor near-duplicates of CTS 1, 2, and 3, although test conditions were not repeated exactly. Test 7, not listed in Table III, was only a "dry run" which involved no fire. Tests 8, 9, and 10 utilized portions of composite barrels as test specimens, with test 8 using a "hot" propane flame as the fire source, and tests 9 and 10 using a JP-5 jet fuel pool fire. Tests 11 and 13 provided even more realism in simulating aircraft crash fires since the test specimens were prototype carbon composite aircraft parts (737 spoilers and an F-16 fuselage section, respectively). Test 12 deviated from realism, at least from the standpoint of civilian aircraft crash fires, by using an explosion to destroy and disperse the burned fiber residue from test 11. However, the failure to execute the explosive phase of the test



as planned (the explosive force was misdirected) minimized the credibility of the results of that test.

### Fiber Detection and Collection Methods

The NWC - China Lake test series utilized a number of proven and unproven techniques for detecting and collecting fire-released carbon fibers. The techniques were separated roughly into passive and active categories. Passive instrumentation was of several general types. "Sticky" paper was a material, either paper or clear plastic film, which had a tacky adhesive coating on one side. This material, which was utilized as 0.2 m x .25 m sheets, 0.15 m by 2.45 m strips, and 0.15 m x 61 m rolls, collected and retained falling carbon fibers when they fell onto the "sticky" paper laid out on the ground or floor. Bridal veil was a fine nylon netting with one-millimeter mesh size. It was mounted vertically, normal to the horizontal flow of air, to catch and retain fibers in the flow of air. The mesh was coated lightly with an adhesive after being mounted on a frame. Among the frames used were "tuna cans" (short, wide cylindrical cans with both ends removed), 0.20 x .24 m vugraph frames, and 2.75 m x 3.0 m wooden frames. Millipore vacuum filters to sample the air were used for early tests, but they generally proved to be unsatisfactory. Relatively large fragments of fibrous carbon residues were collected by simple manual pick-up, not only in the immediate vicinity of the fire tests, but also at distances of up to nearly two kilometers for tests 10, 11, and 13.

Several active types of fiber detection and counting devices were used in the NWC - China Lake test program. The LED device was a light emitting diode source/detector instrument which measured the number rate of fibers passing through its beam for low-level carbon fiber clouds, at ranges of 23 and 91 meters. Micro-wave sensors were developed specifically to measure the mass flux of clusters and clumps of fibers, as well as dense clouds of single fibers. Ball gauge devices, 3.8 cm diameter brass spheres charged with high voltage electricity which were discharged by the close approach of single carbon fibers, not only counted the fibers, but also indicated their lengths. A LADAR (laser detection and ranging) system measured the relative distribution in space of particulate cloud densities at successive times. Infrared imaging systems were used to track the trajectories of hot particulate matter within the fire plume and carbon fiber cloud envelopes. While passive instrumentation supplied most of the fiber count, the active instrumentation contributed a valuable time history of fiber release.

### Test Results

A summary of the NWC - China Lake test results relating to the quantities of fibers released is given in Table IV, while complete

test results can be obtained from reference 10. Not all of the tests were analyzed for carbon fiber release, due to the lengthy times and great expense for counting fibers. However, the thorough analysis of tests 3, 5, 8, 11, and 13 was an adequate representation of the different types of test specimens and conditions.

The amounts of single carbon fibers by mass from burn/explode tests 3 (CTS) and 5 (NWC) were in close agreement (0.010% and 0.008% respectively), indicating the testing and counting techniques were reproducible. However, those amounts of single fibers over 1 mm long were much, much lower than the amounts obtained from the burn/explode tests of propane-burned flat composite plates in the confined chamber at Dahlgren. For example, one typical test in the Dahlgren burn-explode series released 1.78% of the initial carbon fiber present in the composite plate as single fibers, or more than 200 times the quantities from the CTS-3 and NWC-5 tests. NWC-8, involving the propane burn plus explosion of a large barrel specimen, resulted in an equally small detectable release of single carbon fibers. However, when large composite samples (the three spoilers of test 11 and the cockpit fuselage section of test 13) were burned only (no explosive step) in large jet-fuel pool fires, the masses of single carbon fibers released were not too much less (0.0003% and 0.0002%, respectively) than were obtained from two of the propane burn-only tests (0.0008% and 0.0005%) conducted on small samples of the same kinds of spoilers in the confined Dahlgren chamber (see Table II, this report, or ref. 6).

It was conceded generally that the low amounts of single fibers in the NWC - China Lake outdoor tests probably resulted from inadequate sampling procedures. Certainly, in the case of the burn plus explode tests, the carbon fibers could have been propelled to such heights that the resulting clouds of carbon fibers would have drifted well past the limit of instrumentation before the fibers settled, and the amounts released based on the amounts sampled should have been inordinately low. In the case of test 11, the massive smoke plume from the fire was estimated to have exceeded 1000 meters in height, so it is most likely that a great proportion of the single fibers drifted well past the instrumentation limit (2000 meters), since even single ply strips of fibrous matter were transported to 2000 meters.

In spite of the uncertainties in the masses of fibers released in the NWC - China Lake tests, that test series was invaluable to the TRW test personnel, as well as to the NASA investigators, for planning the large-scale, outdoor dissemination and source fire tests conducted by TRW for NASA at the U.S. Army's Dugway Proving Ground, Utah (ref. 12-16). Much of the experience gained in the conduct of the pool fires and in the sampling of the carbon fiber fall-out was beneficial in setting up the Dugway tests. The relatively small (27.5-meter square) Jacob's ladder used at China Lake for mounting bridal veil vugraph fiber collectors was expanded to

a 305-meter square Jacob's ladder used so successfully in the Dugway tests to sample the entire fire plume. The experience gained in the lay-out and collection of passive instrumentation devices (sticky paper and bridal veil) resulted in the smooth, rapid execution of the same tasks during the Dugway tests.

The NWC - China Lake series was successful in several more direct ways. The length spectra of the carbon fibers generated from the burning carbon composites in large, jet-fuel fires confirmed the surprisingly short lengths of fibers released from small-scale, gas-fired laboratory composite burn tests. Furthermore, the observation that many of the fibers released from the burning of actual composite aircraft parts (test 11 spoilers and test 13 cockpit section) had been reduced significantly in diameter (see a later section of this paper on fiber diameters) led to a more intensive study of the generation of small diameter fibers (reference 22). Also, the ability to conduct large-scale fire tests in an unconfined environment with large, thin-skinned composite aircraft parts demonstrated that substantial quantities of fibrous forms other than single fibers (figure 1) were generated from large, realistic fuel fires. Although such forms represent less severe and shorter range electrical threats, investigators were not aware, prior to the China Lake tests, that those species could be lofted to such considerable distances (2000 meters or more). And finally, the mass balances for the large-scale fire tests, while inadequate to define accurately the extent of single fiber release, did confirm a growing suspicion that a substantial amount of carbon fiber mass can be completely consumed by oxidation (see Table IV), thus diminishing the risk of electrical damages.

#### Dahlgren Shock Tube Demonstration Tests

Virtually all of the equipment vulnerability tests for the carbon fiber risk program were performed in relatively small, confined test chambers. The test method (see reference 23, for example) generally involved exposing electrical equipment to quantities of virgin (unused) carbon fibers, all cut to near identical lengths. A persistent question throughout the program was just how representative those clean carbon fibers were of the fibers which would be released from carbon composites burning in a fire of jet fuel, as would occur in civilian aircraft crashes. Not only would the fire-released fibers vary in length, but also the soot, oil fumes, and water vapor and other combustion products could conceivably contaminate the fibers. As a result, the electrical characteristics of fire-released carbon fibers could be envisioned to differ markedly from those of virgin carbon fibers.

To study that possibility, a test was developed which involved the burning of composites in a moderate-sized Jet A (JP-1) fuel fire in a large steel tube at the Naval Surface Weapons Center, Dahlgren, VA. At a point in a long, horizontal steel tube where

the diameter was 4.6 meters, a quantity of small pieces of carbon fiber-epoxy composites was burned in a rotating wire basket over a 1.5 square meter pool fire, until all of the composite had been burned away. During the 2-1/4 hours of the fire, the smoke plume carrying released carbon fibers was pulled through a 275-meter long section of the tube by up to six large fans at the exit end of the shock tube. The fibers in the smoke plume were carried past six identical fan-cooled, operating, stereo amplifiers with known vulnerability levels (to virgin fibers). The smoke plume then passed through a water fog to scrub out the carbon fibers prior to exiting the tube. Complete details of the conduct of all the Dahlgren shock tube tests and the test results are reported in reference 11. However, in summary, the stereo amplifiers failed very close to the expected fiber exposure levels which had been predicted on the basis of tests with virgin carbon fibers with a uniform length spectrum.

In addition to demonstrating the suitability of clean carbon fibers for simulating the vulnerability of electrical equipment to fire-released fibers, the shock tube test also provided a quantitative check on fiber release values from burning composites in an authentic jet fuel fire. A total release, as single fibers, of 0.75% of the mass of carbon fiber initially present in the burned composites was measured. This number was a close check on the 1% single fiber release value used in the risk computations for fire-only crash events. Even the 0.75% release was probably very conservative (over-estimated the hazard), since not only was the burning composite agitated severely in a rotating basket over the fire, but also the composites were divided into very small pieces (which would promote fiber release from the edges) and the fire duration of 2-1/4 hours was excessive for most aircraft crash fires.

#### Dugway Proving Ground Source and Dissemination Tests

A series of large-scale aviation jet fuel fire tests were conducted at the U.S. Army's Dugway Proving Ground, UT. Two of the tests, called "source" tests, were designed specifically to determine the amounts of carbon fibers given off during the burning of relatively large amounts of carbon composite material in outdoor aviation jet fuel fires of such size as to be representative of commercial aircraft crash fires over the past ten years. Three other tests were designed to provide experimental data regarding the predicted dispersion of fibers into the environment, although these "dissemination" tests also supplied information about the quantities of fibers released from the fires.

These large-scale outdoor tests, which are reported in detail in references 12-16, capitalized upon the earlier experience acquired from the TRW outdoor tests conducted at China Lake. As has been pointed out in this report, the favorable performance of 27.5-meter square Jacob's ladder used for the mounting of vertical

sampling devices justified the development of a much larger Jacob's ladder (305-meter square) for use in the Dugway dissemination tests. That larger Jacob's ladder, together with improved active and passive sampling devices and more extensive and distant deposition fiber collectors, resulted in a much more confident assessment of the extent of fiber dissemination.

The two source tests were 10.7-meter diameter fires with 3000 gallons of JP-4 jet fuel. Each fire burned for 1200 seconds, beneath a steel mesh table on which were placed 45-kilogram quantities of carbon-epoxy composite materials. Because the source tests were conducted in essentially zero speed wind conditions, the smoke plume containing released carbon fibers rose directly overhead. Four 60-meter towers supported an overhead array of numerous stainless steel canisters with stainless steel mesh to filter out carbon fibers, as the smoke plume passed through the array of samplers at an altitude of 40 meters.

The three dissemination tests utilized the same types of fires and composite sample arrangements as the source tests, but the wind conditions were required to be approximately 5 meters per second (10 nautical miles per hour) and from a wind direction of  $320^{\circ} \pm 35^{\circ}$ . These conditions were appropriate to direct the smoke plume from a fire through the Jacob's ladder, which contained a variety of sampling devices. Of the three dissemination tests, the smoke plumes for two of them (D-1 and D-2) were only partially intercepted by the Jacob's ladder samplers, while the D-3 plume was centered on the net. The results of the five tests are presented in Table V. The quantities of fibers released, in terms of percentage of total mass of carbon fiber initially present in the burned composites, are quite consistent, considering the differences in wind conditions between the source and dissemination tests. A possible reason for the lower release from test D-3 compared to D-1 and D-2 may be due to the types of composite specimens burned. The pieces in test D-3 were fewer and much heavier than those used for D-1 and D-2. Although the release of up to 0.23% single fibers in the Dugway tests was somewhat higher than the corresponding releases for burn only laboratory tests, the amounts were not surprisingly higher when the differences in fire and sample sizes, fuel types, and wind conditions are considered.

#### CHARACTERISTICS OF FIRE-RELEASED FIBERS

During the early years of the carbon fiber study, vulnerability tests were performed on electrical and electronic equipment using virgin carbon fibers cut into lengths ranging from 5 to 10 millimeters. Those lengths were selected for reasons having no bearing to the true lengths of fibers actually released from burning carbon composites. As NASA's program to determine the quantities of carbon fibers released from burning composites got under way, information about the characteristics of those fibers became available. That

information was to have a considerable impact on the magnitude of the electrical hazards of carbon fibers.

### Fiber Oxidation

Early opinions were that carbon fibers would be virtually non-destructible in fires with peak temperatures in the range of 1800-2200° F. Graphitic materials are known to be extremely resistant to oxidation at much higher temperatures, and carbon fibers have been loosely equated with graphite fibers. However, the assumptions that carbon fibers are the same as graphite fibers and thus cannot be burned up were erroneous. Not only are the high modulus (50-75 million psi) types of carbon fibers (often referred to as "graphite" fibers) far from being true graphite, but also the medium modulus (30-35 million psi) carbon fibers generally used in composites at the present time undoubtedly have less ordered structures than do the high modulus varieties. Consequently, they are much less resistant to oxidation in fires than are the high modulus fibers, to say nothing of true graphite fibers. It soon became apparent that some unknown but substantial portion of carbon fibers exposed to fires could be consumed by oxidation, providing the fire temperatures were sufficiently high, enough air was present, and/or the composite was burned for an adequate period of time. Early composite burn/explode tests in the Dahlgren test chamber must have been too mild in terms of the above requirements for significant fiber oxidation to have occurred, but later laboratory tests (ref. 7) demonstrated clearly that large proportions of carbon fibers could be burned away, if the above fire conditions were met. Massive oxidation of carbon fibers was also believed to have been responsible for a large amount of carbon fiber loss during the burning of an experimental composite aircraft cockpit section in a large outdoor jet fuel fire (ref. 10). However, absolute proof was lacking since fiber collection methods during the China Lake tests were probably considered inadequate. The existence of flame temperatures (to 1500 K or 2250° F) and oxygen concentrations sufficiently high to bring about substantial carbon fiber oxidation, were determined in the NASA-White Sands experimental jet fuel fire tests (ref. 17), discussed later.

The fact that there are marked differences in the inherent thermal stabilities of different types of carbon fibers was demonstrated (ref. 6) in a striking fashion when a 0.45 kilogram spool of 30+ million psi modulus carbon fiber, after being burned for 20 minutes with a propane flame, continued to smolder after removal of the flame until more than 90% of the mass of fiber had been consumed. Similar treatment of a 50+ million psi modulus fiber led to self-extinguishment of the fire when the flame source was removed, with the resultant total consumption of a negligible amount of fiber mass by oxidation.

Since almost all of the carbon fiber used now, and expected to be used in the future, in civilian aircraft composite structures is of the medium (30-35 million psi) modulus type, most of the fiber release testing was done with composite built from the two most common fibers presently in use (T300 and AS). Therefore, the conclusions in this report about the physical characteristics of fire-released fibers may be valid only for medium modulus fiber composites. The amount of oxidation, the fiber lengths and diameters, and perhaps even the weight percentages and numbers of released fibers from burned composites from high modulus carbon fibers may be at variance with the results of this report.

There may well be differences in the thermal behavior of different makes of fibers of the same general class (such as the medium modulus class) and even between new and old versions of the same fiber from the same manufacturer, depending upon the amounts of impurities present. It has been known that the thermal durability of carbon fibers at 588 K (315°C) was highly dependent upon the sodium impurity: the greater the sodium content, the greater the weight loss (ref. 24). Indeed, independent proposals to eliminate the electrical risks from carbon fibers released from burning composites have been based on the concept of adding certain metallic ion impurities to the fibers to catalyze, or promote, their complete oxidative consumption in fires (ref. 25, 26). This impurity-catalysis effect was used to explain some earlier anomalous test data, and the report also emphasized the importance of the state of carbon fiber purity on the ultimate physical dimensions of fire-released fibers (ref. 22).

#### Fiber Lengths

The early realization that carbon fibers released from burning composites were much shorter than had been expected was welcomed. That finding inferred a lessening of the possibility that the fibers would cause electrical problems, since it was generally agreed that short fibers could bridge far fewer electrical gaps than could long fibers. Initially, released carbon fibers were counted only when they were over one millimeter in length, since that length was agreed upon as the lowest fiber length which might cause electrical problems, and there had to be some limitation on the amount of testing of the vulnerability of electrical equipment. A summary of the length categories (over 1 mm) resulting from a large number of fiber release tests are given in Figure 11 and it is apparent that the greatest number, from one-half to three-quarters of those released, fell in the 1-2 mm length range. With very few exceptions, the mean lengths for fibers released in tests were less than 3 millimeters.

It was not until well into the risk analysis program that it was found that the large majority of fire-released carbon fibers were shorter than one millimeter, a fact that diminished even

further the numbers of electrical gaps or circuits that could be affected (Table VI). The proportions of those fibers under one millimeter ranged as high as 87-98% of the mass of fiber released for composites which were not only burned but also destroyed by an explosion (Tests CTS-1, -2, and -3, ref 12), but were more realistically from 67-74% of the fiber mass for composites which were burned (Test BT-244 and DL-39) with no subsequent destructive force being applied to the fibrous residue (ref. 6). Disturbing the fibrous residue with a moderate flow of air (tests AF-4 and AF-6) released somewhat longer fibers, but the majority were still less than 1 millimeter in length. Even more dramatic was the fact that in all cases, more than 90% of the numbers of fibers were shorter than 1 mm., and therefore, virtually harmless in an electrical sense.

The inherent tendency of state-of-the-art carbon fibers to be emitted in such short lengths may be due to the preferential, rapid oxidation of numerous sites along the length of the fiber. One study (ref. 22) showed, via scanning electron microscopy, that large numbers of developing pits and flaws are present in partially oxidized carbon fibers (figure 12); such flaws could be generated at sites with relatively high concentrations of metallic ion impurities (e.g., sodium) which could have been entrapped within the acrylic precursor to the carbon fibers.

#### Fiber Diameters

The process of oxidation of carbon fibers not only led to a majority of short fibers and complete consumption of much of the fibrous material in the fire, but was also expected to lead to a certain amount of incompletely oxidized fibers which would be apparent by their reduced diameters. The burning of actual composite structural parts in large jet fuel fires in the China Lake test series confirmed that expectation (ref. 10). Those structural parts which showed a reduction in diameter, typified by the spoiler in figure 13, had relatively thin composite skins. In contrast, the 6.4-mm thick composite plate burned with a propane flame in test CTS-3 resulted in very little fiber diameter reduction compared to the spoiler fibers. However, flat, 3.2 mm thick composite plates burned with propane and exposed to a steady 15 m/sec (30 knot) flow of air released fibers with even smaller diameters, about 2 microns, versus the original 7.5 to 8 micron diameters ( see figure 14). Therefore, it must be concluded that given the proper fire conditions, that is, suitably high temperatures (1800° F or more) and available air supply to the flame, carbon fibers can be oxidized easily either completely or to very fine fibers.

#### Potentially Respirable Fibers

The possibility of formation of carbon fibers with such small dimensions as to be respirable by humans, with some as yet non-existent harmful physiological effects the result, raised a concern



beyond the potential for electrical damages from the use of carbon fiber composites in civilian aircraft. Under the Intergovernment Committee Action Plan, the National Institute for Occupational Safety and Health (NIOSH), on behalf of the U.S. Public Health Service, was assigned the responsibility for assessing any future health implications relating to the carbon fiber study. However, since NASA had done most of the fire-induced fiber release testing, it was appropriate for NASA to be aware of the generation of any fibers which would be of interest to NIOSH.

Exploratory work at Langley (ref. 20) showed that at least a small amount (~6%) of the total number of single carbon fibers collected from a jet fuel burn of composites were of such small dimensions as to be potentially respirable, that is, with diameters less than 3 micrometers (microns) and lengths less than 80 microns. Table VI lists 3 separate tests which produced small amounts of such fibers. A contractual effort was then initiated to better quantify the amounts of such sized carbon fibers which would be released from various composite fire events. The results of that extensive investigation (ref. 22) established the fact that under certain conditions, for example, with relatively thin composites and/or a turbulent fire, substantial numbers of potentially respirable fibers (in comparison to the numbers of fibers over one millimeter in length) could be emitted from burning carbon composite aircraft parts in jet fuel fires. The average sizes were 1.5 microns in diameter and 30 microns in length. The formation of fibers with small diameters was attributed not only to the simple oxidation of fibers from their original 8 micron diameters, but also to a "fibrillation" phenomenon involving the mechanical shattering of brittle, burned carbon fibers into smaller, thinner fragments.

Only one investigation of the effects of carbon fibers on test animals is known to have been reported (ref. 27). That work involved relatively short term exposure of guinea pigs to carbon fiber "dust" produced by mechanical abrasion. Although the concentrations of carbon fiber particles were suitably high in comparison to asbestos fiber concentrations, very few of the particles which resulted from the milling action had the requisite dimensions to be considered respirable (less than 3 microns diameters, length-to-diameter ratios from 3:1 to 10:1, and lengths less than 80 microns). Therefore, the potential short term effects should still be considered uncertain.

In the absence of any evidence that carbon fibers of any size could or could not, have adverse health effects on humans (except for the cutaneous allergic reactions so typical of many fibrous substances), comparisons were made to the quantities of concern in the case of asbestos fibers. Using the conditions and some of the results of one of the large-scale, outdoor composite burn tests at Dugway Proving Ground as the scenario for a simulated, extreme case aircraft crash-fire, a maximum concentration of respirable-sized

carbon fibers which was only one-half the OSHA-allowable ceiling concentration for asbestos fibers was computed. Furthermore, the total exposure to those carbon fibers from the accidental crash-fire was predicted to be less than 0.01 the OSHA-allowable 8-hour exposure to asbestos fibers. Field tests conducted by NIOSH during NASA's Dugway Proving Ground dissemination tests confirmed the very low amounts of very small size carbon fibers which were released by burning carbon composites in large jet fuel pool fires (ref. 14). Those results showed that the mass of carbon fibers of potentially respirable size which were sampled was more than 40 times the mass of fibers which were of electrical concern (over 1 mm long). However, based on an average exposure to respirable-sized fibers for the three dissemination field tests of  $0.04 \times 10^6$  fiber-seconds per cubic meter, the average concentration of those small fibers was only 0.4% of the maximum allowable by OSHA in the asbestos workplace. Furthermore, the total exposure to the small fibers over the 20-minute duration of each test was less than 0.1% of the total exposure to asbestos fibers which would be allowed by OSHA for an 8-hour day.

#### Other Fibrous Residues

The great majority of the fiber release studies was directed toward quantifying the extent of single fiber release. Likewise, virtually all of the vulnerability testing of electrical equipment was conducted with single fibers. The greatest electrical threat was considered to be from single fibers, since they would not only be dispersed to the greatest distances, but also would penetrate interfaces such as doors, filters, and equipment cases easier than would other types of fiber residues. In addition, the initial fiber counting procedure at the Dugway Proving Ground was based on a statistical technique which was valid only for single fibers. Nevertheless, a number of other forms of carbon fibers, pictured in figure 1, were generated by burning composites. Laboratory tests which used the relatively gentle gas flame led to a preponderance of single fibers. The augmentation of the flame effects by disturbing the burning or burned composites with explosions, air flow, impact, or even forces internal to the residual fiber "biscuit", such as twisting and flexing actions, created varying amounts of clusters (lint), strips, and fragments. Clusters of several to hundreds of single fibers agglomerated together probably resulted from "snow-flaking" in the air from regions of extremely high fiber concentration. The strips of fibers appeared to originate from the separation of areas of single plies of carbon fiber tape in crossplied laminates. And the fragments, which were much thicker than strips, were formed when severe disturbing forces (e.g., explosions, air blasts, and impacts) were applied to the burned-out composites.

Laboratory fire tests supplied only a limited amount of data relating to the generation of multi-fiber residues. The results of the burn/explode tests conducted in the Dahlgren chamber (ref. 4, 5)

included a very crude mass balance separating residues into a hand pickup of large fragments, a broom sweep of single fibers, lint, and small fragments, and a vacuum fraction of very small particles and carbon dust. The AVCO fire test data reported in reference 7 included single fibers, lint, and occasional very small fragments which were transported by the moderate air flow through 2-3 meters of the test chamber. And the drop impact data generated in the Scientific Services, Inc. test series (ref. 8) included the total fragments produced in addition to the single fiber efflux.

The first thorough investigation of not only the quantities of multi-fiber particulate matter, but also their distances of dispersion, was performed by TRW for the China Lake test series (ref. 10). Clusters of fibers accompanied the single fibers to the extent of sampling (about 2000 meters), although both fiber forms were very sparse at that distance. Strips of fibers, which were easy to locate since they were up to 5 centimeters wide and as long as one meter or more, were also lofted to the 2000 meter limit of collection. An excellent accounting of strip size and location throughout the test area was included in reference 10. Thick fragments from explosions were limited to less than 100 meters immediately surrounding the fire. The comprehensive accounting for the total efflux from several of the China Lake tests included, in addition to the location of the multi-fiber species on the test range and their physical dimensions, but also the vertical distribution of all particulates on a Jacob's ladder collecting net and the mass of each type of fiber matter recovered.

An even more accurate accounting of the various types of fibrous materials given off by burning actual composite aircraft parts under realistic conditions was accomplished by the TRW group during the large scale aviation jet fuel fire tests at Dugway Proving Grounds (ref. 12-16). The collection scheme at Dugway profited by TRW's earlier experience at China Lake. The much larger test site permitted the collection of lofted particles for distances up to 19 kilometers, in contrast to the 2 km. limit at China Lake. Furthermore, the much larger Jacob's ladder (305-meter square) at Dugway (compared to the 30-meter square size at China Lake) permitted a much improved sampling of the smoke plume carrying the fibers.

#### FIRE PLUME AND CHEMISTRY STUDIES

Surprisingly little useful information about the nature of aviation jet fuel fires was available at the beginning of the carbon fiber risk program. Some analytical modeling of methane and natural gas fires had been done, more recently by NASA-Ames Research Center, but experimental verification of the codes was lacking. Furthermore, the methane and natural gas models were inadequate substitutes for kerosene-based fuel, including aviation jet fuels, which create exceptionally sooty, oily smoke plumes.

The only known experimental work with such fuels involved fires too small to apply to many aircraft crash fires.

A program to provide models for suitably large aircraft crash fires, together with the necessary fire dynamics and chemistry data for experimental verification of the models, was conducted under the direction of the Chemical Projects Office at NASA's Ames Research Center. Such a confirmed model was felt desirable for understanding the release of carbon fibers from the lower regions of a fire, the transport of the fibers from the fire to the upper reaches of the fire plume, and the extent of consumption by oxidation of the fibers. In order to predict the fiber behavior, a number of detailed characteristics of large fuel fires had to be determined.

#### Science Applications, Inc. Analysis - Phase I

The first phase of the analytical modeling of large, liquid fuel fires by Science Applications, Inc. (SAI) resulted in a mathematical analysis, based on the best operational data available at the time (ref. 28). The model included the predicted profiles of flame velocities and temperatures, as well as the expected spatial variations in fire chemistry, including fuel and oxygen distributions. Several assumptions involving the physical phenomena of the large pool fires had to be made because of the paucity of experimental data. Consequently, some uncertainties affecting the oxidative consumption of carbon fibers, a key objective of the study, were identified. These were, (1) the spatial distribution of temperature and available oxygen (oxygen not involved in fuel burning), and (2) the dependence of the rate of carbon fiber surface oxidation on temperature and available oxygen. Subsequently, two experimental programs were conducted to reduce the uncertainty of those factors. One was a series of tests carried out at NASA's White Sands (NM) Test Facility to measure the fire dynamics and chemistry of large, liquid pool fires. The other program was an extensive laboratory thermogravimetric analytical study at NASA's Langley Research Center to quantify the rates of oxidation of carbon fibers.

#### NASA White Sands Jet Fuel Pool Fire Tests

Three large, well instrumented JP-4 pool fires were conducted at White Sands (ref. 17). The tests were designed and managed by NASA-Ames and carried out by White Sands personnel. Pool fires with 7.62-meter (one fire) and 15.24-meter (two fires) diameters were run, at essentially zero wind conditions. Above each fire was an array of instrumentation at various points along the radii of the fires, and at heights ranging from 0.72 meters (2.35 feet) up to 22.8 meters (70 feet). The instrumentation at those points measured temperatures and major constituent species in the fire, including oxygen, nitrogen, hydrogen, water, carbon monoxide, carbon dioxide, argon, methane, hydrocarbons (fuel) and soot. Only limited

flame velocity measurements were achieved, due to heat-caused mechanical difficulties. The results of this series of fire tests, relative to the SAI Phase II modeling effort, are described in the following section.

#### Science Applications, Inc. Analysis - Phase II

The White Sands study showed that the actual maximum flame temperatures were well below those originally predicted by their Phase I model. The lower than expected temperatures were found to be the result of incomplete mixing of fuel and air, and thus, only partial consumption of fuel. Temperatures no higher than 1500-1600K (figure 15) were measured, in contrast to predictions up to 2400K. However, oxygen concentrations were in reasonable accord with projected values. The use at each point in the flow of the flame of a fluctuating chemistry model (ref. 29), which assumed a certain proportion of the oxygen to be unavailable for fuel combustion, served to reduce some of the error due to "unmixedness" of fuel and air. Still, the peak predicted temperatures of 1800-2000K were considerably higher than were observed experimentally.

With regard to the consumption of carbon fibers by surface oxidation, the NASA Langley thermogravimetric study (see following section) revealed significantly lower rates of oxidation than predicted with the original SAI model (ref. 38). Analysis by SAI of the empirically-derived factors (low temperatures and unavailable oxygen) revealed that a relatively small mass fraction, typically 15 percent, of the mass of a fiber is oxidized when the fiber is released in the lower region of a large fire and is transported above the fire by the vertical flow of the fire plume. However, in addition to the surface oxidation of the fiber when released, it is likely that the process of oxidation may commence well before a given fiber is released from a composite which is exposed to the fire. Fibers temporarily attached at or near the surface of a composite may be extensively or even completely oxidized if retained at that site for tens of seconds.

#### Thermogravimetric Analysis of Carbon Fibers

The aforementioned thermogravimetric analysis (TGA) study of carbon fibers carried out at NASA Langley provided information on rates of oxidation of carbon fibers at temperatures prevalent in fires. Although such data was available for the thermal aging of carbon fibers at much lower use temperatures (up to 573 K), none had been reported for the high temperatures representative of liquid fuel fires. The Langley experiments were conducted up to 1250K (1787°F), which was not only the maximum attainable with the equipment on hand, but also was considered to be a representative jet fuel fire temperature.

The TGA tests were performed by heating carefully weighed quantities of carbon fibers in a sensitive micro-balance at a rapid

rate up to 1250K. After that temperature was reached, the fiber was heated isothermally and the mass loss was monitored until it was completely consumed by oxidation. Since a fire environment does not have a uniform or constant air or oxygen content, the tests were run in several different gas environments: air (21% oxygen), 8.16% oxygen, 4.18% oxygen, and pure nitrogen. Some of the results of the TGA study are given in figure 16. The AS carbon fiber, which is typical of the types of carbon fibers used extensively in fabricating aircraft composite parts, was completely consumed in an air environment in less than one minute, but it resisted significant degradation in the completely inert nitrogen environment. Between those two extremes, the fiber oxidized away at rates markedly less than in air. In an actual jet fuel fire, the entire range of oxygen content would undoubtedly exist for varying periods of time and in different regions of the fire. The information generated by this study was used to advantage by Science Applications, Inc. to determine predicted carbon fiber oxidation rates, which were so important for their fire modelling effort.

#### FIBER DETECTION AND COUNTING STUDY

As was pointed out in the section on outdoor fiber release tests at China Lake, CA., most of the single fiber detection and collection was with passive methods. And by far the same was true for all of the laboratory fiber release testing. However, a consequence of the passive techniques was the necessity to count the fibers by optical microscopic procedures which were slow, expensive, and subject to variations related to the individuals doing the work. A task to develop a more reliable and rapid counting method was delegated to the Jet Propulsion Laboratory (JPL).

Exploratory experiments at JPL showed that a high voltage spark method to detect carbon fibers was promising for adaptation to a fiber counting and measuring system. Although the principle behind the technique was far from being unique, JPL incorporated several key changes which tailored the method specifically for carbon fibers (ref. 9). Because of the high resistance of most carbon fibers (3000 ohms/cm or more), a carbon fiber draws only a very small current between a pair of grid electrodes under a moderately high voltage bias (500 to 2000 volts), enough perhaps to heat the fiber red-hot, but not enough to burn it up rapidly. Generally, the accumulation of fibers on an electrified grid decreases the sensitivity of the grid. JPL overcame that problem by using a pulse discharge technique, involving the storage of energy in a capacitor. Thus, repeated pulsation was effective in completely consuming the carbon fibers.

For all practical purposes, fire-released carbon fibers of electrical concern were found to be between 1 and 5 mm long (see figure 11). Therefore, the JPL investigators combined five independent (not in series) grids with spacings to count fibers in the

1-2, 2-3, 3-4, 4-5, and 5-10 mm length ranges. Each grid had an independent pulse integrator electronic package to shape the pulses for the input spark signals and to integrate those pulses to provide a DC voltage output corresponding to the total number of fiber counts. The resulting signal was then recorded on a strip chart recorder. The total assembly utilized a stainless steel, square mesh pre-screen to prevent clusters of fibers from short-circuiting the grid-bias circuitry. A fan and windbox arrangement provided the air sample.

Although the design of the system was not optimized, a number of important parameters of the operation were studied. Among them were: the voltage of the high voltage discharge, the capacitance of the discharge unit, airflow rate, depth and shape of the grid electrodes, lengths of fiber fragments, and effect of fiber clusters.

While the development of the JPL counting system was not accomplished in time to be utilized to its full potential in the carbon fiber risk analysis program, several successful trial applications of the prototype device demonstrated the practicality of the concept. The device was used at the top of a combustion chimney to detect fibers released from burning composites in JPL alternate materials experiments (ref. 25). Equally successful counting of released fibers in a small NASA Ames laboratory composite burn chamber was also demonstrated. In a large-scale test check-out, the detector successfully monitored the release of carbon fibers in a Dahlgren shock tube composite burn test (ref. 11). The results, which agreed closely with those from an independently-developed electrified grid detector (ref. 30), helped show that the fiber capture capability of sticky tape collectors in the smokey jet fuel fire plume may be low. Later experiments at the Jet Propulsion Laboratory proved that soot generated in kerosene-fueled fires did not lead to the generation of spurious signals with the JPL high voltage spark device.

Although active devices can provide dynamic fiber information, such as fiber flux values, the passive sticky paper collectors can be used readily and more economically at many separate locations, and then stored for later counting. A second task performed by the JPL team was to modify the high voltage spark system for the counting of carbon fibers which had been collected on passive sticky paper collectors (ref. 31).

A specialized high voltage discharge system was built to "read" sticky paper samples upon which single fibers had been captured. A single grid with a 1.19-mm spacing and operating under a bias of 510 volts was the heart of the tape-reading system. When placed onto the sticky paper holding the captured carbon fibers, a rapid spark discharge from four parallel high voltage capacitors caused the consecutive burn-up of carbon fibers on the tape. The rate of discharge could be varied at will from 5 to 100 Hz, depending on

the fiber density. Calibration of the device with standardized tape samples established the average of multiple discharges necessary to burn out each fiber (for fibers of uniform length deposited in random fashion) at about 2-1/2 per fiber.

Although this system was not completely optimized and tested, the progress was sufficient for the investigators to achieve a statistical understanding of spark counts initiated by carbon fibers on a sticky paper medium, using a high voltage grid system. Sufficient data was obtained to establish a calibration factor for counting the carbon fibers on a specific sticky paper surface. Prototype hardware was developed and demonstrated in laboratory testing.

Further development of the concept could probably lead to its adaptation to the counting of carbon fibers collected on the surfaces of filters, such as the micropore types. Fiber length distributions could also be obtainable by using several suitably-spaced grid networks. Also believed possible is the adaptation of the concept to the counting of sub-millimeter sized particles of importance for environmental considerations.

#### CONCLUDING REMARKS

An extensive program involving all aspects of the release of carbon fibers was conducted. The principal objective of this investigation was to assist in the prediction of amounts and characteristics of carbon fibers which would be generated from the crash and burning of civil aircraft having structural parts made from carbon fiber-reinforced epoxy materials.

Hundreds of composite-burning experiments were conducted on a laboratory scale. A number of parameters which could affect fiber release were studied, including the types and degree of disturbance applied to the burning composites, and nature of the composite itself, and the type and proportions of fuel. The disturbance applied to the burning composite was shown to be the most critical factor influencing single fiber release, leading to very low amounts of fiber based on the amount initially present in the burned composite for burning alone, to somewhat greater release when the burned composites were subjected to explosive destruction (fig. 17).

Large scale aviation jet fuel fires were conducted to demonstrate the validity of the laboratory fire-release experiments. The burning of up to 45 kilogram quantities of carbon fiber composite aircraft parts in 15-meter diameter pool fires confirmed the generally low level of release of carbon fibers in laboratory tests, with less than a quarter of a percent of single fibers being given off. Demonstration tests also showed that the electrical characteristics of fire-released carbon fibers were equivalent to those of virgin carbon fibers which neither had been combined with matrix resins nor had been exposed to fires.



The release of unexpectedly small amounts of carbon fibers from burning composites was attributed in part to the fact that carbon fibers generally used in state-of-the-art aircraft composites can be consumed extensively by oxidation in aviation jet fuel fires. Empirical evidence of oxidative consumption in fires was supported by a laboratory thermogravimetric analytical study, as well as by fire plume dynamics and chemistry studies.

For the purpose of risk computations, a figure of 1% of the mass of carbon fiber originally present in burned aircraft composite parts was recommended for the 85% of those civilian aircraft crashes expected to involve fire alone (fig. 18). Three and one-half percent of the fiber was anticipated to be released in fires having an explosion. These single fiber release values reflected only those fibers of concern in an electrical sense, that is, over one millimeter long. Furthermore, the fibers predicted to be released were assumed to be of an exponential length distribution (for those over 1 mm long), with a mean length of 2 millimeters.

#### REFERENCES

1. A Report of Observed Effects on Electrical Systems of Airborne Carbon/Graphite Fibers. NASA TM 78652, 1978.
2. Carbon Fiber Study. NASA TM 78718, 1978.
3. Bell, V.L.: Source of Released Carbon Fibers. NASA Conference Publication 2074, 1979, pp 41-71.
4. Babinsky, T.C.; and Musselman, K.A.: Burn/Blast Tests of Aircraft Structural Elements. U.S. Naval Surface Weapons Center, NSWC/DL TR 3897, 1978.
5. Babinsky, T.C.; and Musselman, K.A.: Burn/Blast Tests of Miscellaneous Graphite Composite Parts. U.S. Naval Surface Weapons Center, NSWC TR 79-390, 1979.
6. Babinsky, T.C.: Fiber Release From Impacted Graphite Reinforced Epoxy Composites. U.S. Naval Surface Weapons Center, NSWC TR 80-216, 1980.
7. Alexander, J.G.: Development of a Fire Test Facility for Graphite Fiber-Reinforced Composites. (AVCO Corporation; NASA Contract NAS1015511). NASA CR 159193, 1980.
8. Wilton, C.; Kamburnoff, G.; and Boyes, J.: Fire Testing of NASA Samples - Phase I. (Scientific Services, Inc., NASA Contract NAS2-9945). NASA CR 152339, 1979.
9. Yang, L.C.: High Voltage Spark Carbon Fiber Detection System. Jet Propulsion Laboratory Publication 80-30, 1980.
10. Lieberman, P.; Chovitt, A.R.; Sussholz, B.; and Korman, H.F.: Data Reduction and Analysis of Graphite Fiber Release Experiments. (TRW Defense and Space Systems Group; NASA Contract NAS1-15465). NASA CR 159032, 1979.
11. Pride, R.A.; McHatton, A.D.; and Musselman, K.A.: Electronic Equipment Vulnerability to Fire Released Carbon Fibers. NASA TM 80219, 1980.
12. Pride, R.A.: Large Scale Carbon Fiber Tests. NASA TM 80218, 1980.
13. Chovit, A.R.; Lieberman, P.; Freeman, D.E.; Beggs, W.C.; and Millavec, W.A.: Carbon Fiber Plume Sampling for Large Scale Fire Tests at Dugway Proving Ground. (TRW Defense and Space Systems Group; NASA Contract NAS1-15465). NASA CR 159215, 1980.

14. Chovit, A.R.; and Freeman, D.E.: Data Tabulation from Carbon Fiber Plume Sampling for Large-Scale Fire Tests at Dugway Proving Ground. (TRW Defense and Space Systems Group; NASA Contract NAS1-15465). NASA CR 159216, 1980.
15. Whiting, J.H.; Magann, G.N.; Peterson, W.A.; and Sutton, G.: Large-Scale Outdoor Fire-Released Carbon Fiber Experiments. U.S. Army Dugway Proving Ground, DPG-FR-80-301, 1980.
16. Peterson, W.A.; and Carter, F.L.: Methodology Study for the Development of a Passive Sampler for Fire-Released Carbon Fibers. U.S. Army Dugway Proving Ground, DPG-TR-78-314, 1980.
17. Lindley, L.: Measurements of the Spatial Dependence of Temperature, Gas and Soot Concentrations Within Large, Open Hydrocarbon Fuel Fires. NASA White Sands Test Facility TR-259-001, 1980.
18. Pride, R.A.: Carbon Fiber Counting, NASA TM 80117, 1980.
19. Review and Analysis of Fiber Counting Methods. U.S. Naval Surface Weapons Center, NSWC TR-80-210, 1980. (Arthur D. Little, Inc.).
20. Bell, V.L.: Release of Carbon Fibers From Burning Composites. NASA Conference Publication 2119, 1980, pp. 29-57.
21. Taback, I.: Evaluation of Equipment Vulnerability and Potential Shock Hazards. NASA Conference Publication 2119, 1980, pp. 73-99.
22. Sussholz, B.: Evaluation of Micron Size Carbon Fibers Released from Burning Composites. (TRW Defense and Space Systems Group; NASA Contract NAS1-15465). NASA CR 159217, 1980.
23. Newcomb, A.L.: Carbon Fiber Exposure Test Facility and Procedures. NASA TM 80220, 1980.
24. Gibbs, H.; Wendt, R.C.; and Wilson, F.C.: Carbon Fiber Structure and Stability. 33d Annual Technical Conference, 1978; Reinforced Plastics/Composites Institute, The Society of Plastics Industry, Inc.
25. Ramohalli, K.: Novel Approaches for Alleviation of Electrical Hazards of Graphite-Fiber Composites. Jet Propulsion Laboratory Publication 79-63, 1979.
26. Reardon, J.; and Russell, L.: Metal Ion Catalyzed Combustion of Carbon Fibers and Resin Matrices. 14th Carbon Conference, Pennsylvania State University, June 25-29, 1979.

27. Holt, P.F.; and Horne, M.: Dust From Carbon Fiber. *Environmental Research*, 17, 1978, pp. 276-283.
28. Analytical Prediction of Atmospheric Plumes and Associated Particle Dispersal Generated by Large Open Fires. (Science Applications, Inc.; NASA Contract NAS2-10039). NASA CR 152337, 1978.
29. Harsha, P.T.; Bragg, W.N.; and Edelman, R.B.: Preliminary Report: Improvement of a Mathematical Model of a Large Open Fire. (Science Applications, Inc.; NASA Contract NAS2-10327). NASA CR 152338, 1979.
30. Experimental and Analytical Studies for the NASA Graphite Fiber Risk Assessment. (The Bionetics Corporation, NASA Contract NAS1-15238). NASA CR-159214, 1980.
31. Yang, L.C.; and Hull, Glen L.: High Voltage Spark Carbon Fiber Sticky Tape Data Analyzer. Jet Propulsion Laboratory Publication 80-55, 1980.

TABLE I. - CARBON FIBER RELEASE TEST FACILITIES

Laboratory Testing

NASA Langley Research Center  
Navy's NSWC Dahlgren (VA) Chamber  
AVCO Corporation Fire Test Facility  
Scientific Services, Inc. (Redwood City, CA) Facility  
Jet Propulsion Laboratory

Demonstration and Large-Scale Testing

Air Force/TRW at NWC China Lake, CA  
Navy's NSWC Dahlgren Shock Tube Facility  
Army's Dugway Proving Ground, UT

Fire Plume and Chemistry Testing

NASA White Sands Test Facility

TABLE II. SINGLE CARBON FIBERS (CF) RELEASED FROM TESTS OF  
SPECIMENS FROM COMPOSITE AIRCRAFT PARTS

36

←←← Uncorrected →→→						←←← Corrected →→→		
Part and test no. <sup>a</sup>	Part no. <sup>b</sup>	Wt. CF in g. <sup>c</sup>	Length (ave.) ,mm	No. of fibers (Dugway count)	% CF <sup>d</sup>	Correction factor <sup>e</sup>	No. of fibers	% CF
<u>737 Spoiler</u>								
BT-119/X-89	1	64	3.0	19.9 x 10 <sup>6</sup>	8.1	--	--	--
BT-120/X-90	2	69	6.5	48.0 x 10 <sup>6</sup>	40.6	x .35	16.8 x 10 <sup>6</sup>	14.2
BT-122/X-92	4	69	4.2	28.3 x 10 <sup>6</sup>	15.5	x .35	9.9 x 10 <sup>6</sup>	5.5
BT-124/X-94	6	65	2.6	27.7 x 10 <sup>6</sup>	10.0	x .35	9.7 x 10 <sup>6</sup>	3.5
BT-126/X-96	13	46	4.6	33.2 x 10 <sup>6</sup>	30.0	x .35	11.6 x 10 <sup>6</sup>	10.4
BT-129/X-99	9	178	3.2	44.1 x 10 <sup>6</sup>	7.1	x .35	15.4 x 10 <sup>6</sup>	2.5
BT-244	1	64	1.8	1.91 x 10 <sup>4</sup>	0.0048	--	--	--
BT-245	2	69	1.9	.34 x 10 <sup>4</sup>	.0008	--	--	--
BT-246	9	178	2.1	.45 x 10 <sup>4</sup>	.0005	--	--	--
<u>DC-10 Rudder</u>								
BT-103/X-73	--	79	2.5	26.3 x 10 <sup>6</sup>	7.4	x .35	9.21 x 10 <sup>6</sup>	3.6
BT-104/X-74	--	372	2.9	98.9 x 10 <sup>6</sup>	7.0	x .35	34.6 x 10 <sup>6</sup>	2.5
BT-105/X-75	--	325	3.3	161 x 10 <sup>6</sup>	14.7	x .42	56.5 x 10 <sup>6</sup>	6.2

<sup>a</sup>BT - /x- indicates a composite burn followed by explosion; BT- indicates burn only.

<sup>b</sup>See reference 4

<sup>c</sup>Estimated

<sup>d</sup>% CF =  $\frac{\text{no. fibers counted} \times \text{ave. length (cm)} \times \text{wt. in g. of 1 cm of fiber } (9 \times 10^{-7})}{\text{wt. of carbon fiber in sample}} \times 100$

<sup>e</sup>See reference 19. Correction factor of 0.35 extended down to 25 million count.

TABLE III. - MATRIX OF TESTS AT NWC<sup>a</sup> CHINA LAKE

Test no.	Test location <sup>a</sup>	Type sample	Type fuel <sup>b</sup>	Burn time (sec)	Explode? <sup>c</sup>	Complete analysis?
1	CTS	Flat plate	Propane	2400	Yes	Yes
2	CTS	Flat plate	Hot propane	1200	Yes	Yes
3	CTS	Flat plate	Hot propane	1200	Yes	Yes
4	NWC	Flat plate	Propane	1200	Yes	No
5	NWC	Flat plate	Hot propane	1200	Yes	Yes
6	NWC	Flat plate	Hot propane	1200	Yes	No
8	NWC	Barrel	Hot propane	1200	Yes	Yes
9	NWC	Half barrel	JP-5 <sup>d</sup>	540	No	No
10	NWC	Half barrel <sup>e</sup>	JP-5	300	Yes	No
11	NWC	Spoilers (3)	JP-5	240	No	Yes
12	NWC	Spoiler Debris	JP-5	420	Yes	No
13	NWC	Cockpit	JP-5	360	No	Yes
14	NWC	Flat Plate	Propane	1200	Yes	No
15	NWC	Flat plate	Hot propane	1200	Yes	No

<sup>a</sup>NWC - Naval Weapons Center; CTS - Capistrano Test Site

<sup>b</sup>Propane: 1900-2000°F flame; hot propane: 2300-2400°F flame

<sup>c</sup>Fifty-seven grams (2 ounces) of C-4 explosive, except CTS-3 (114 grams)

<sup>d</sup>Plus 1.36 kilograms of aluminum and magnesium

<sup>e</sup>Plus burned residue from test no. 9

TABLE IV. - CARBON FIBER MASS BALANCES FOR NWC CHINA LAKE  
TESTS

Test no.	Type sample	Initial Fiber mass, g. (est)	Total deposition <sup>a</sup>		Single fiber		% Fiber unaccounted <sup>b</sup>
			mass, g.	% initial mass	mass, g.	% initial mass	
3	Plate	611	168	19.2	0.08	0.01	--
5	Plate	611	105	12.0	.07	.008	--
8B <sup>c</sup>	Barrel	3,690	--	--	--	--	84
8E <sup>c</sup>	(Residue)	< 575	50	.7	.49	.007	79
11	Spoilers(3)	2,630	3	.08	.01	.0003	29
13	Cockpit	16,200	9	.04	.04	.0002	76

<sup>a</sup>Single fiber, clusters, fragments 1 to 20 mm long

<sup>b</sup>Includes fibers dispersed but not collected, fibers shorter than 1 mm, and fibers consumed by oxidation

<sup>c</sup>Burn only - 8B; exploded residue - 8E



TABLE V. - SINGLE FIBERS WITH LENGTHS GREATER THAN 1 mm  
RELEASED IN DUGWAY JET AVIATION FUEL FIRE TESTS

Test no.	Carbon fiber mass in fire, kg <sup>a</sup>	Average length of single fibers, mm	Single fibers released		
			Number	Mass, g	% of initial mass
D-1	32.34	3.2	$3.1 \times 10^8$	64	0.20
D-2	31.84	3.1	3.5	72	.23
D-3	49.54	3.2	2.4	51	.10
S-1	34.01	3.3	2.9	63	.19
S-2	31.88	3.2	2.2	46	.14

<sup>a</sup>Carbon fiber mass calculated as 0.7 composite mass

TABLE VI. - SIZES OF CARBON FIBERS FROM COMPOSITE  
BURN TESTS

Test <sup>a</sup> Number	Test Type	Ave. length L, mm	Length < 1 mm		Respirable size <sup>b</sup>	
			Wt. %	No. %	Wt. %	No. %
CTS-1	Burn/explode	0.05	86	99.9	--	--
CTS-2	Burn/explode	.05	84	99.6	--	--
CTS-3	Burn/explode	--	98	>99.9	--	--
BT-244	Burn	.30	67	95	--	C
DL-39	Burn	.33	74	95	0.25	6.4
AF-4	Burn + 15 m/sec	.35	63	94	.17	5.6
AF-6	of air	.40	53	92	.28	12.1

<sup>a</sup>CTS - Capistrano Test Site; BT - Burn Test (Dahlgren Chamber);  
DL - Dahlgren Shock Tube; AF - Air Flow (Dahlgren Chamber)

<sup>b</sup>"Respirable" includes fibers less than 3.5-4.0 micrometers  
in diameter and with length: diameter between 3:1 and 10:1

<sup>c</sup>Incomplete analysis indicated less than 0.6% of the fibers  
had diameters less than 3.5  $\mu$ m. No L:D available.

SINGLE FIBERS  
 SIZE: 1 TO 8  $\mu$ m DIA. 0.1 TO 15 mm LONG  
 FALL RATE: 2 cm/sec  
 DISPERSION RANGE: 0 TO ~100 km



CLUSTERS OR LINT  
 HUNDREDS OF FIBERS  
 FALL RATE: 10 - 20 cm/sec  
 DISPERSION RANGE: 0 TO 10 km



STRIPS  
 SINGLE LAMINAE: 0.15 mm THICK, VARYING  
 LENGTHS AND WIDTHS  
 FALL RATES: 1 TO 5 m/sec  
 DISPERSION RANGE: 0 TO 2 km



IMPACT FRAGMENTS  
 MULTIPLE LAMINATE PIECES  
 OCCURS ONLY IN IMMEDIATE VICINITY  
 OF CRASH/FIRE

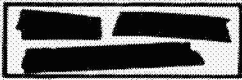


Figure 1.- Carbon fiber residues released from burning composites.

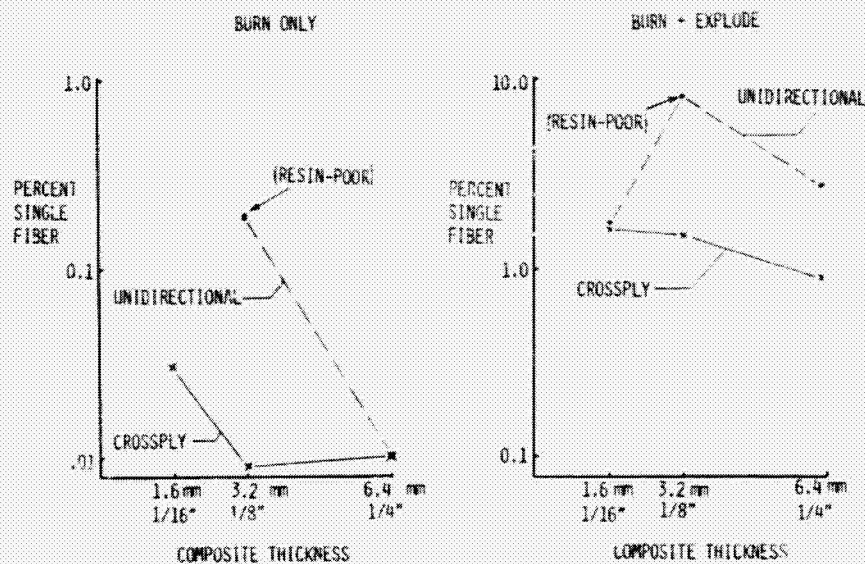


Figure 2.- Effects of composite configuration on single fiber release.

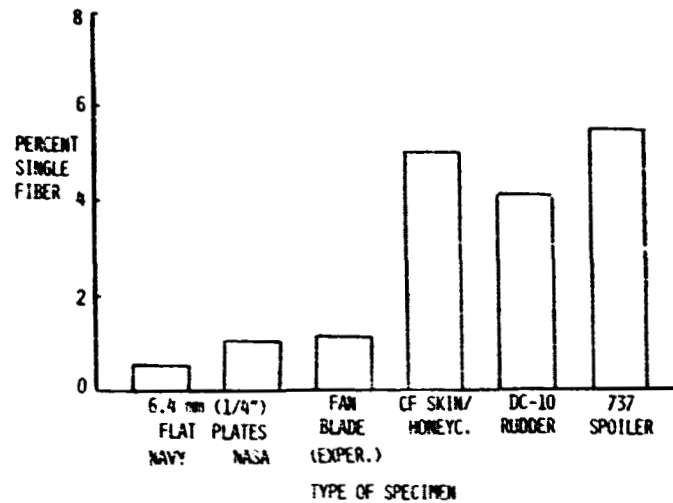


Figure 3.- Single fiber release from prototype composite aircraft parts exposed to fire plus explosives.

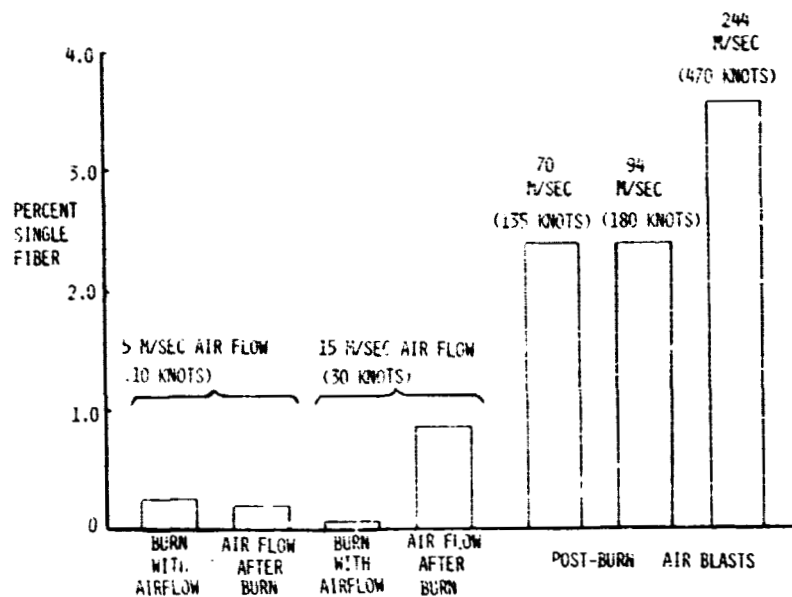


Figure 4.- Effects of high and low airflow on single fiber release.

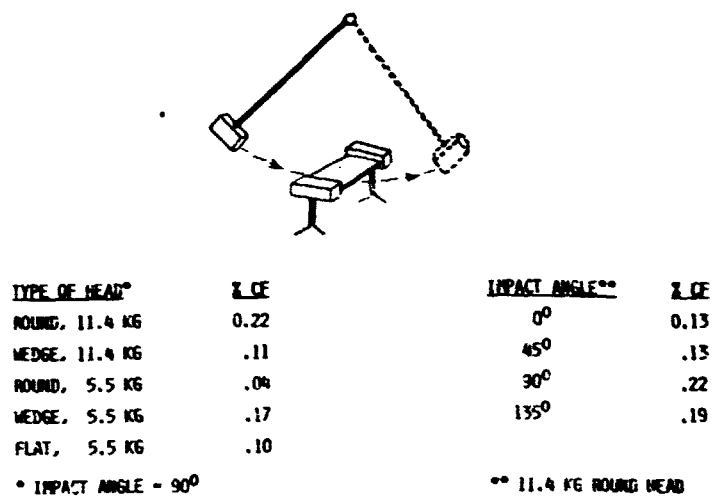


Figure 5.- Effects of pendulum impact on single fiber release.

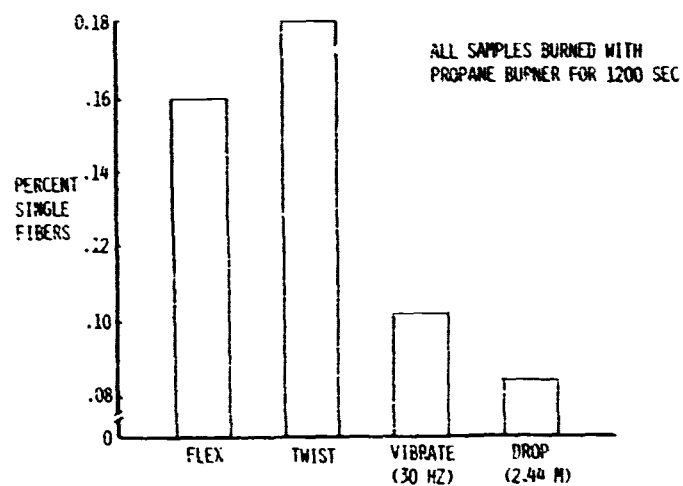


Figure 6.- Effects of internal disturbances on single fiber release.

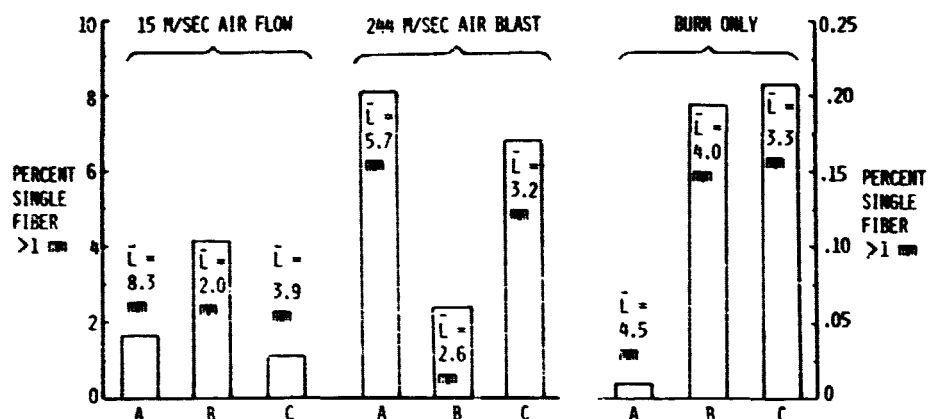


Figure 7.- Release of single carbon fibers from carbon fiber/NOMEX honeycomb panels under different test conditions. A-Panel skins: 2 plies of carbon tape ( $0 + 90^\circ$ ) with fiberglass scrim. B-Panel skins: 2 plies of carbon fabric. C-Panel skins: 1 ply of carbon fabric and 1 ply of Kevlar fabric.  $\bar{L}$  = mean fiber length.

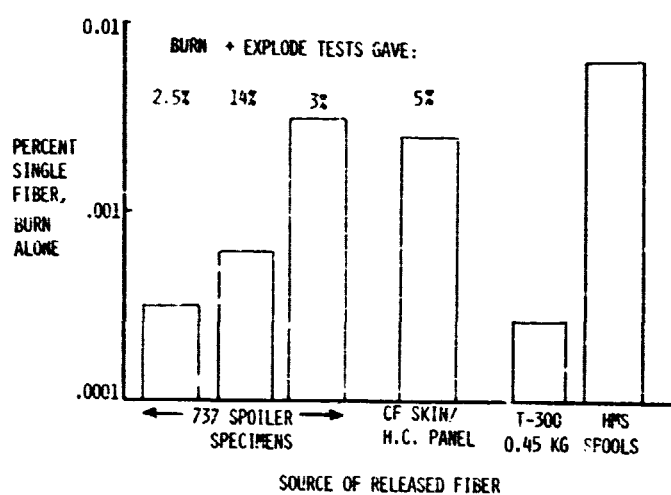


Figure 8.- Release of single carbon fibers from propane fire only and fire/explosive tests.

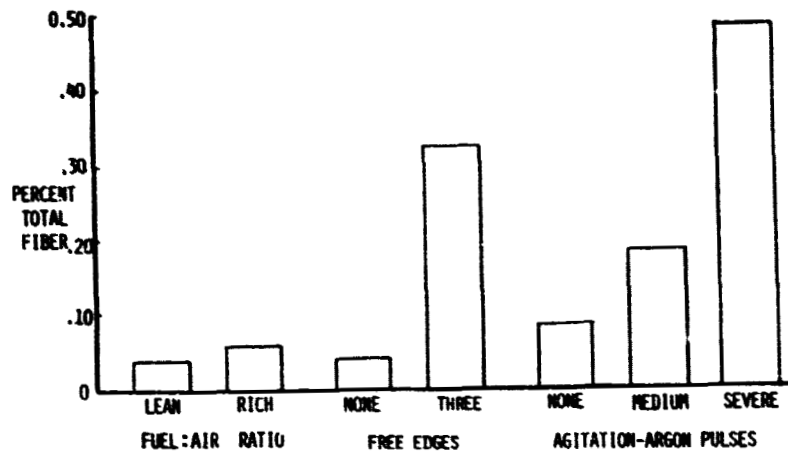


Figure 9.- Effects of fire variables on total carbon fiber release.

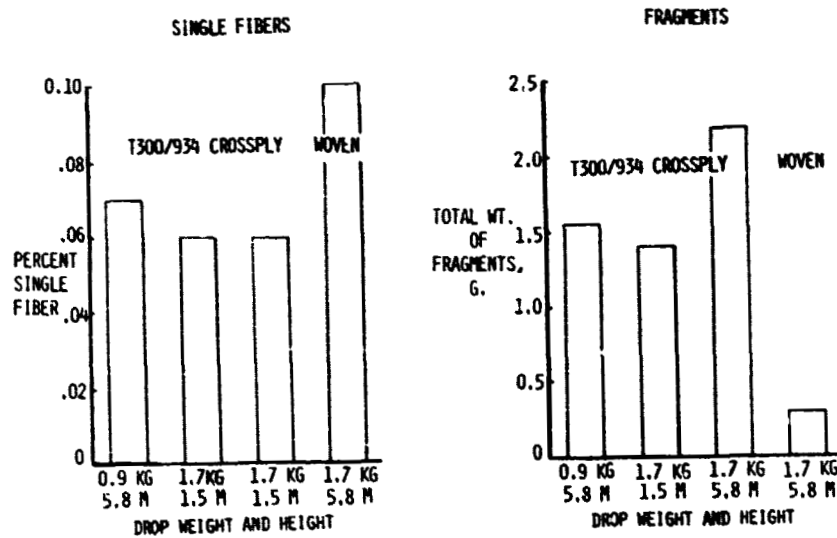


Figure 10.- Single carbon fibers and composite fragments from fire plus drop impact tests.

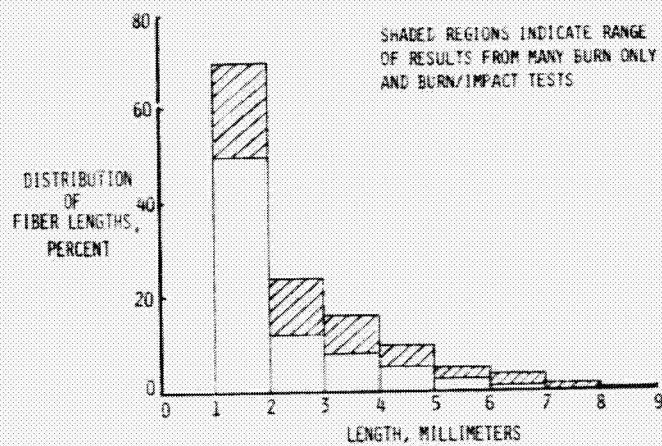


Figure 11.- Spectrum of single fibers over one millimeter long from numerous test procedures.



Figure 12.- Scanning electron photomicrograph of irregularly burned carbon fiber.



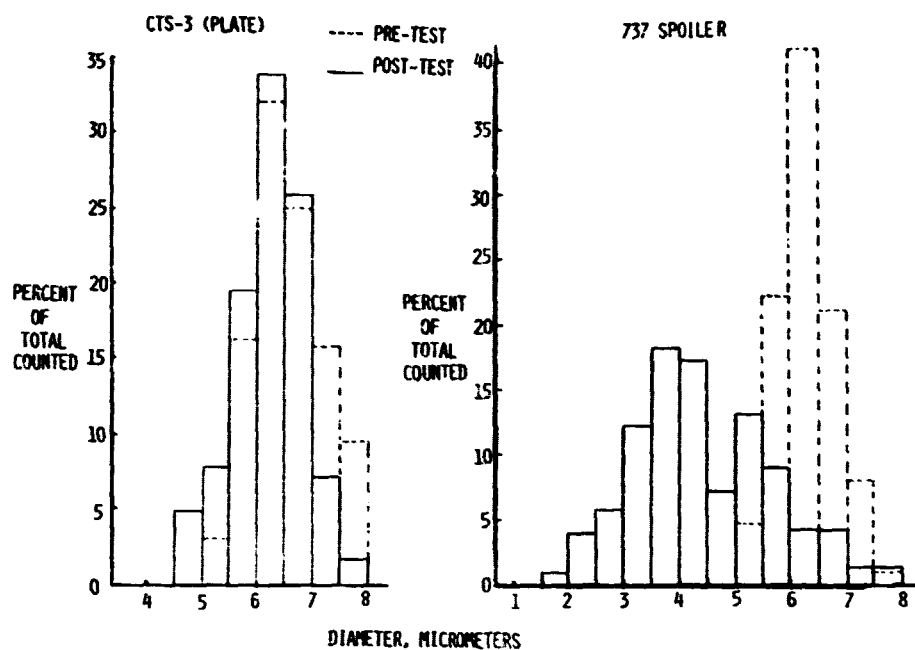


Figure 13.- A comparison of pre- and post-test diameters of carbon fibers.

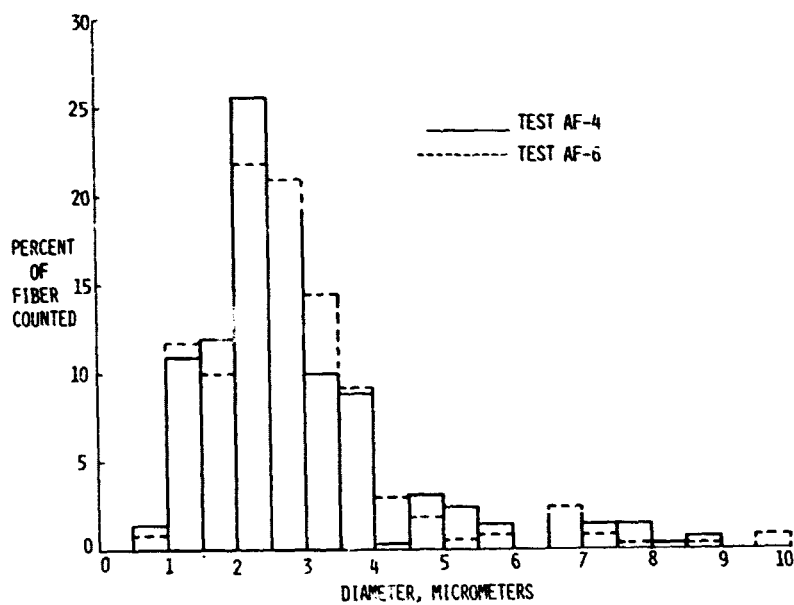


Figure 14.- Spectrum of reduced fiber diameters for two airflow assisted burn tests.

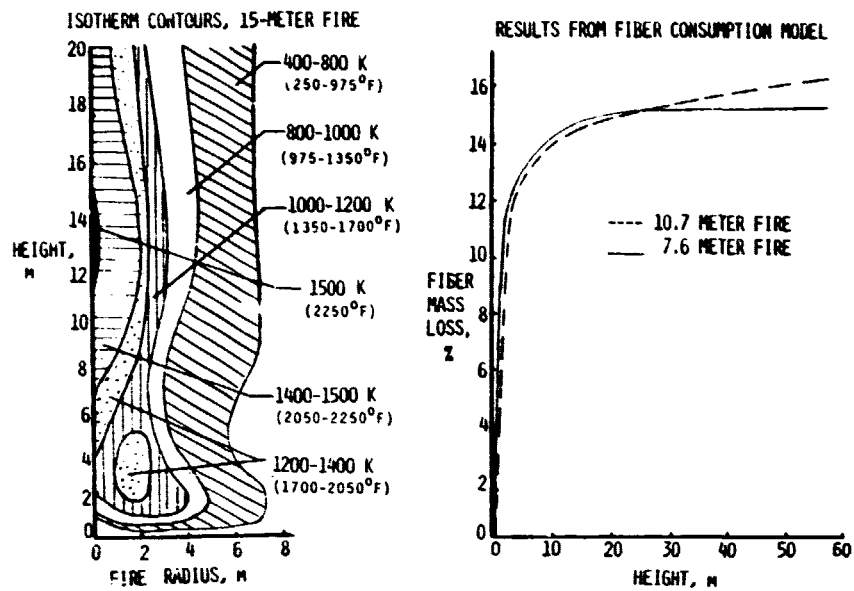


Figure 15.- Results from NASA White Sands fire experiments.

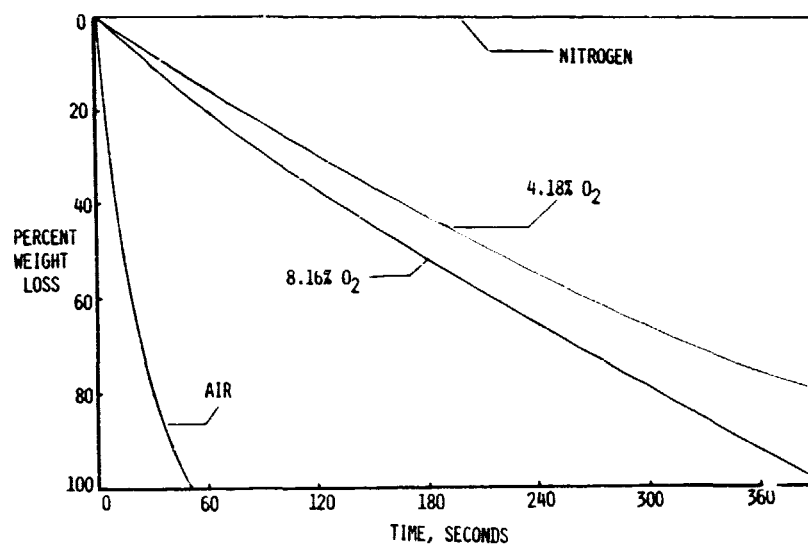


Figure 16.- Iso-thermogravimetric analyses of "AS" carbon fibers.

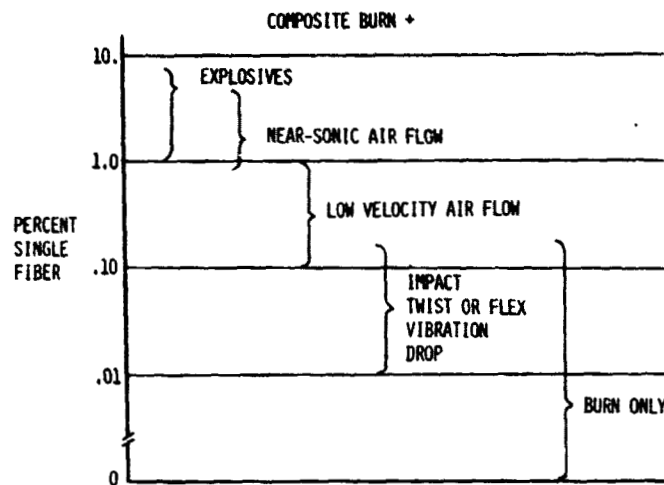


Figure 17.- Summary of effects of disturbances on single fiber release from composites.

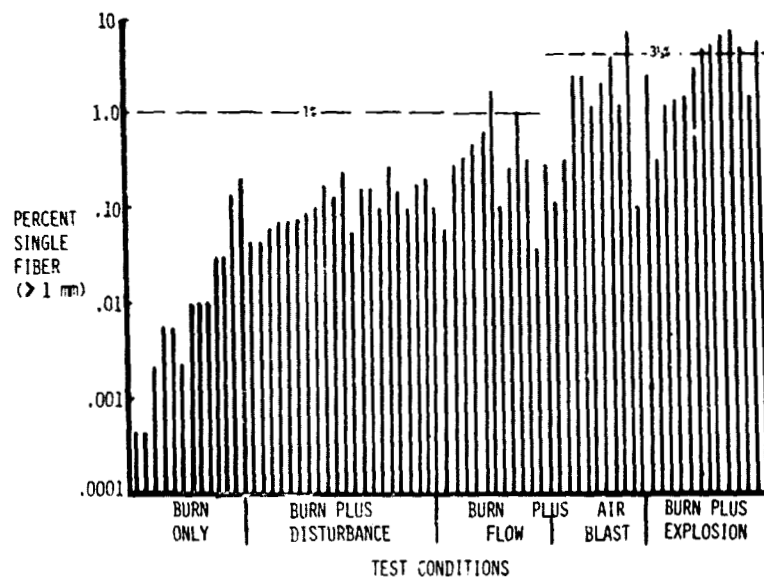


Figure 18.- Summary of numerous laboratory carbon fiber release tests with quantities recommended for risk computations.